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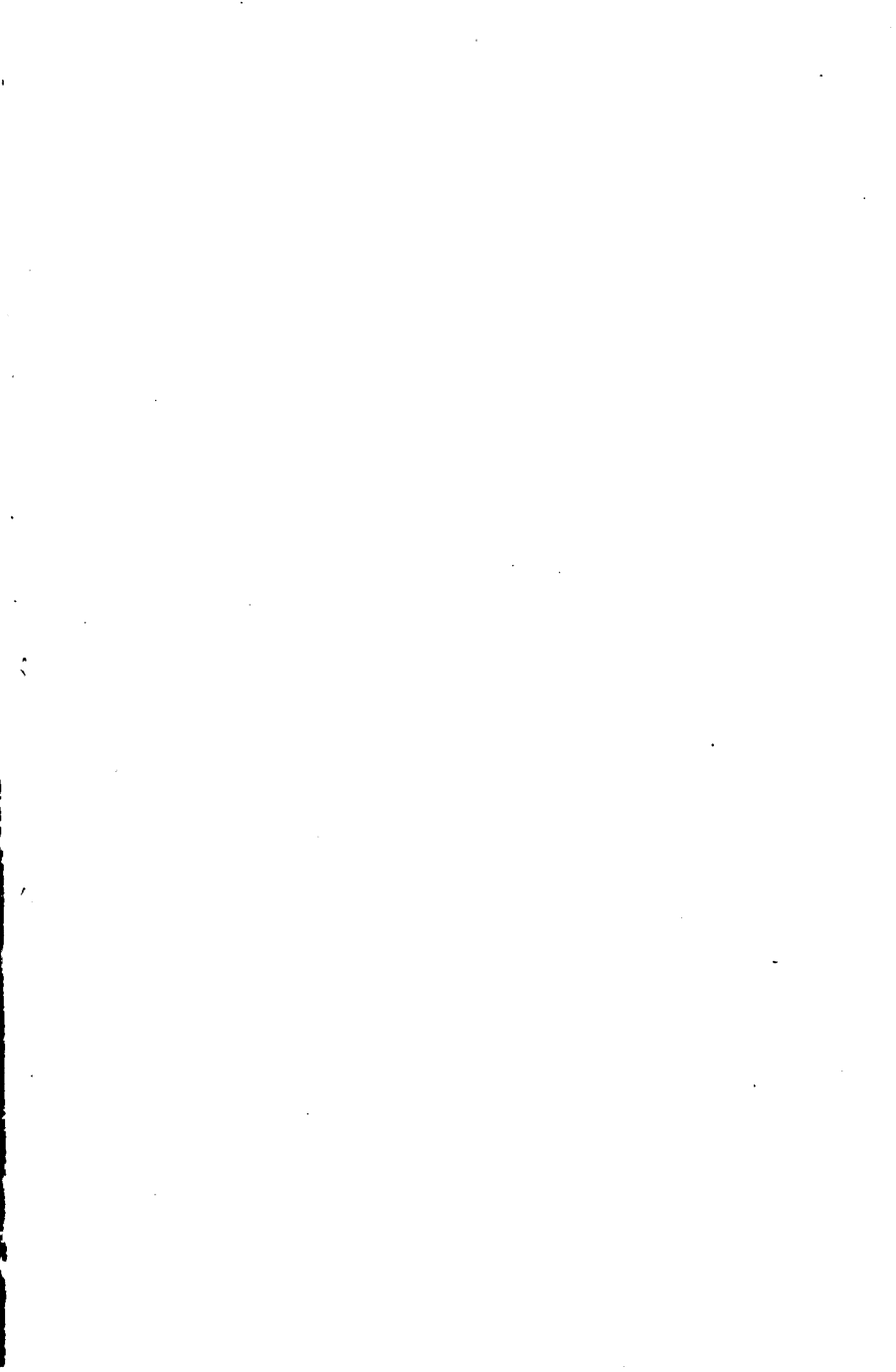
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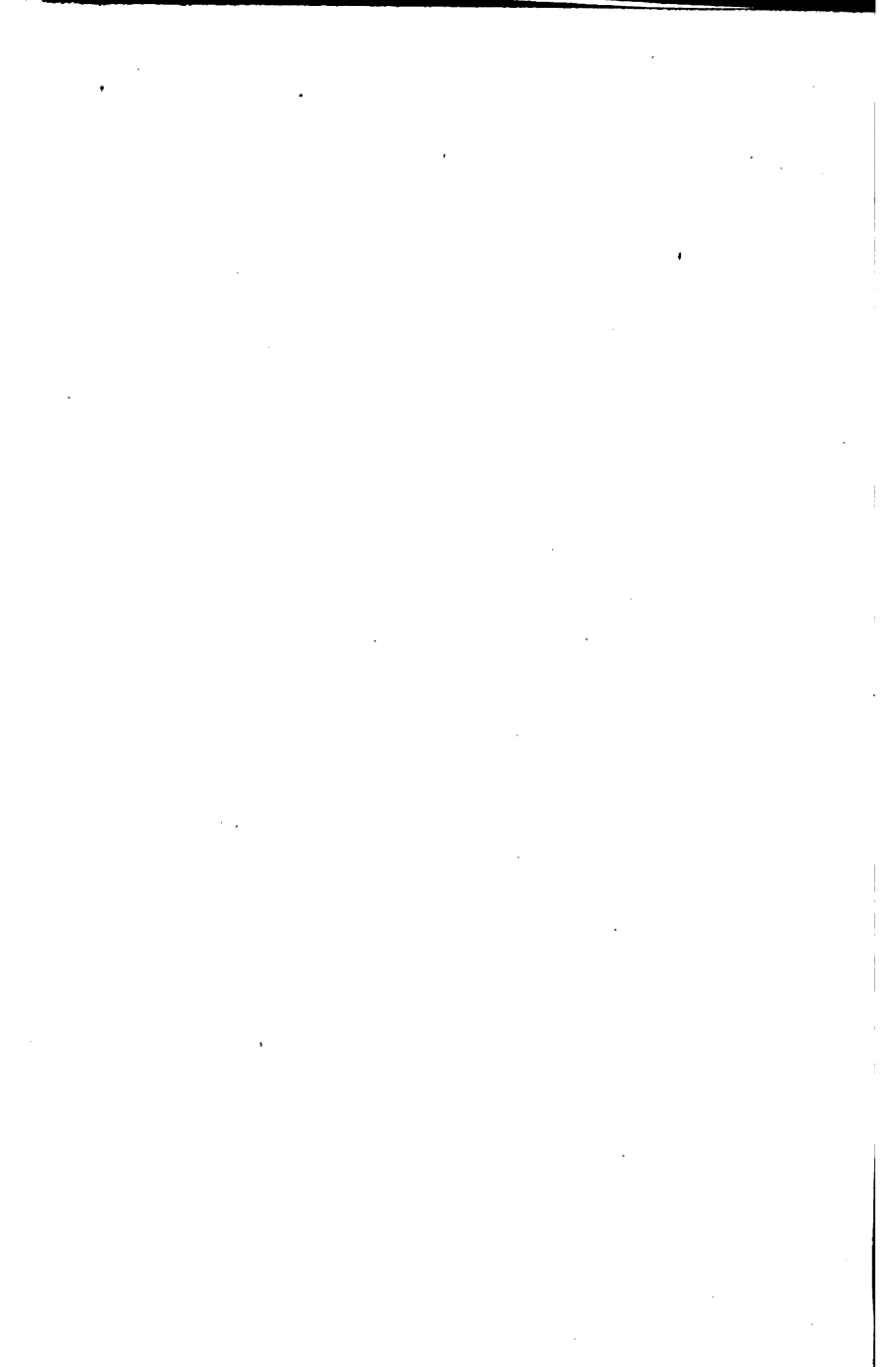


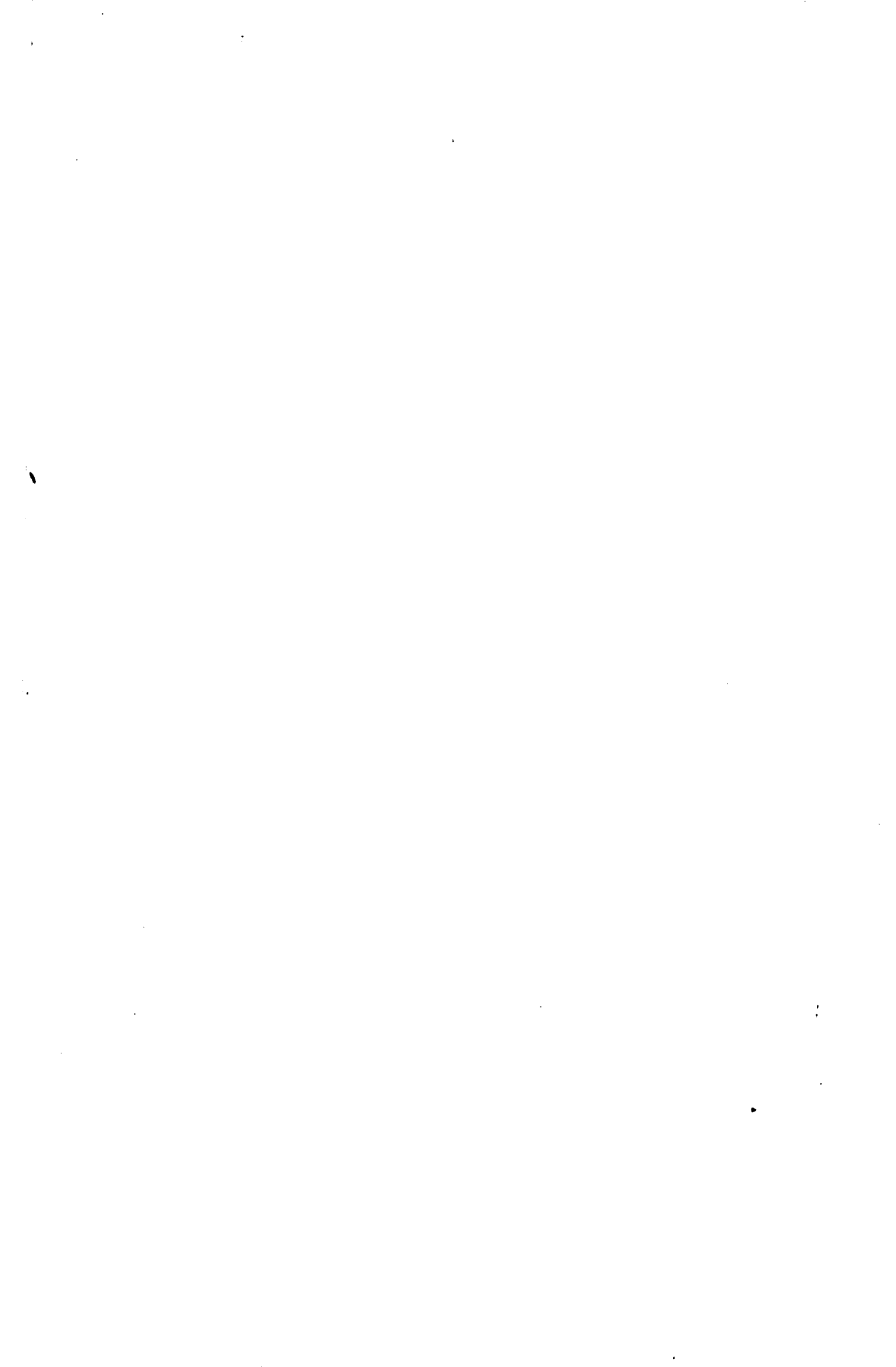
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THE CYANIDE PROCESS

*AN INTRODUCTION TO THE CYANIDE PROCESS,
INCLUDING THE DETERMINATION OF THE
APPLICABILITY OF THE PROCESS
TO AN ORE*

BY

ALFRED S. MILLER

FORMERLY PROFESSOR OF MINING, METALLURGY, AND GEOLOGY
UNIVERSITY OF IDAHO

Author of A Manual of Assaying

SECOND EDITION, REVISED AND ENLARGED
FIRST THOUSAND



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GENERAL

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PREFACE TO THE SECOND EDITION.

As stated on the title page, this book is intended, as "an introduction to the cyanide process, including the determination of the applicability of the process to an ore." It was written primarily for the convenience of the writer's students in doing laboratory work, and the first edition was printed for the writer. The demand from most of the gold-mining regions of the world for copies of this book has made it necessary to issue a second edition.

A mining engineer, after receiving a copy of the first edition, wrote, "It is certainly the most compact and 'to the point' book on this subject that is printed." If the writer has succeeded, even in a measure, to write a "compact" and "to the point" book on this subject, it should be welcomed by all who are not already proficient in this subject.

Figures 9 and 10 are from photographs of laboratory work. Figures 14, 15, and 16 are taken from the catalogue of the Hendryx Electro Cyanide Company. The other illustrations are taken, by permission, from the catalogue of the Pacific Tank Company.

Moscow, ИДАНО, February, 1906.

PREFACE TO THE FIRST EDITION.

A NUMBER of books on the cyanide process are on the market. Articles on this subject constantly appear in the proceedings of scientific associations, and in the mining and metallurgical journals. It might, at first thought, appear unnecessary to publish another book on this subject, especially since books cannot keep up with the progress of such a subject. But there are many mining men and students, who, on account of their limited knowledge of chemistry, and of the practical operations of the cyanide process, are not able to receive much benefit from the publications mentioned above.

This book has not been written for the expert, or for the man who "knows it all"; but for the larger number of persons who have no knowledge of this subject, and are looking for a book that will give them a start, and sufficient of the fundamental principles to understand the literature on this subject. It is also intended to serve the purposes of a syllabus and a laboratory guide.

Clearness was aimed at even at the expense of repe-

tition. Conciseness is essential to attain the object sought in this book.

This being largely a chemical subject, technical terms and chemical equations could not be altogether avoided; nor would it have been desirable to do so.

Acknowledgment is due the Pacific Tank Company for kindly loaning the writer the cuts used in illustrating this article.

MOSCOW, IDAHO, January, 1903.

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THE CYANIDE PROCESS.

INTRODUCTORY.

THE first use of potassium cyanide to dissolve gold in ores, on a commercial scale, is of comparatively recent date. McArthur and Forest took out a patent on the cyanide process in England in 1887, and in the United States in 1889. It was some years after this before a success was made of the process, and not until a success had been made of the process elsewhere did it come into use in America.

That gold is soluble in potassium cyanide was known for a long time. Hagan mentioned it in 1806. Dr. Wright used gold-cyanide solution in electroplating in 1840. (Elsner published his observations in 1844, claiming that the solution of the metals is due to the action of the oxygen absorbed from the air, which "decomposes part of the cyanide.")

J. H. Rae took out the first patent in the United States in 1867, for the extraction of gold by a "current of electricity and suitable solvents or chemicals, such, for instance, as cyanide of potassium." This was

followed by other patents from time to time; but none of these processes came into general use, until McArthur and Forest took out their patents. ~~~~~

The process was developed by many experimenters, and practical workers of the process. In many instances the same facts have been discovered independently by workers in different parts of the world. This makes it impossible to credit everything that is known of this subject to the person that may think he deserves such credit. As this is largely a chemical subject, much that is known depends on the general principles of chemistry, which are the common property of all.

CHAPTER I.

GENERAL DESCRIPTION OF THE CYANIDE PROCESS.

(A DILUTE solution of potassium cyanide dissolves gold and silver contained in crushed ore, and, with some exceptions, does not attack, to a large degree (during the time necessary to dissolve a large percentage of the gold), any of the other constituents of the ore. The gold and silver are then precipitated from the solution, and, after proper treatment, cast into bars. This constitutes what is known as the "Cyanide Process.")

Different methods have been devised by which these results are attained. A general idea of the working of this process may be obtained from Fig. 1, which is a laboratory plant by which about 100 lbs. of ore can be treated at a time. A potassium cyanide solution of the best strength* is put into the uppermost tank. The ore, crushed to the best size,* is put into the next lower tank. Above the bottom, this tank has a perforated false bottom which is covered with duck filter-cloth. The solution, from the uppermost tank, enters the tank containing the ore from the bottom. After the ore has soaked the best length of time,* the gold-

* As determined by experiment.

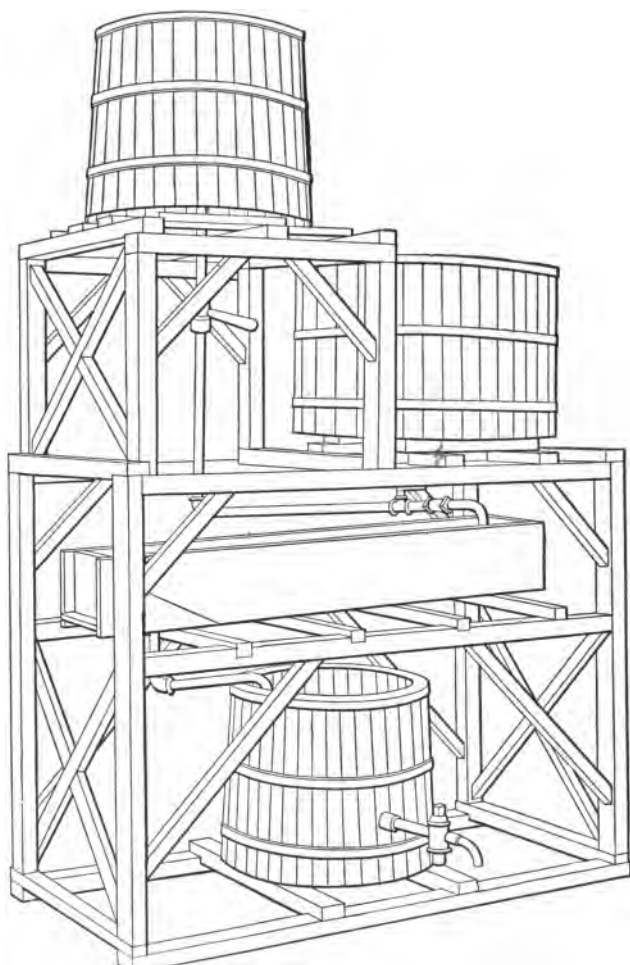


FIG. 1.—Laboratory Cyanide Plant.

bearing solution * is drawn off from below, and passed through the rectangular wooden box below. This box is divided into six large and six small compartments. The solution flows down through the small compartments and up through the large compartments (for arrangement of box, see Fig. 2). Each large compartment is fitted with a wire screen, near the bottom, above which the compartment is filled with zinc shav-

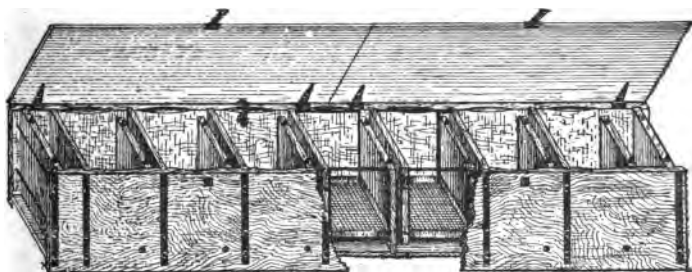


FIG. 2.—Wooden Zinc-boxes.

ings. The zinc precipitates the gold and silver from the solution, and the solution runs down into the lowest, or sump tank. From this tank the solution is poured back (pumped back in large works, see Fig. 5) into the uppermost tank, enough strong solution is added to bring the used solution to the strength it had before it was put on the ore, the ore in the next lower tank is replaced by fresh ore, and all the other operations are repeated as before.

* Generally, the first solution of potassium cyanide is followed by a weaker solution, or a weak solution is used first, followed by a stronger solution; and the last potassium cyanide solution is followed by a water-wash.

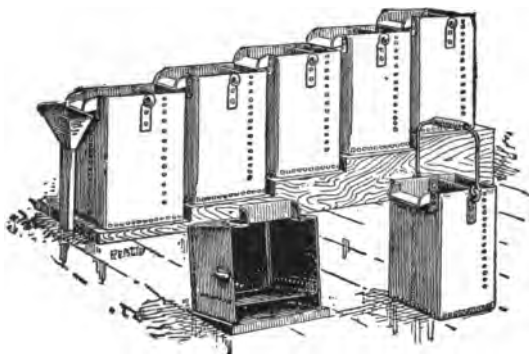


FIG. 3.—Iron Zinc-boxes.

These are light rectangular, sheet-steel boxes, absolutely water-tight and thoroughly coated with acid-proof paint.

In service the boxes are arranged in series as shown. Each box has two compartments and the solution passes down the narrow and up through the large compartment in which the zinc shavings are placed, and overflows into the next box.

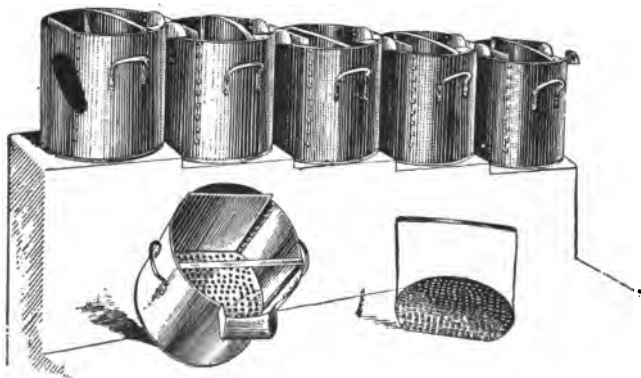


FIG. 4.—Improved Iron Zinc-boxes.

It will be noticed that the box is made round instead of square; this avoids the necessity of filling out corners. Each compartment has a capacity of one cubic foot of shavings. The iron is thoroughly protected by acid-proof paint and every box is fitted with a perforated iron shavings-tray. Each box forms one compartment, and generally six or more compartments are used in series.

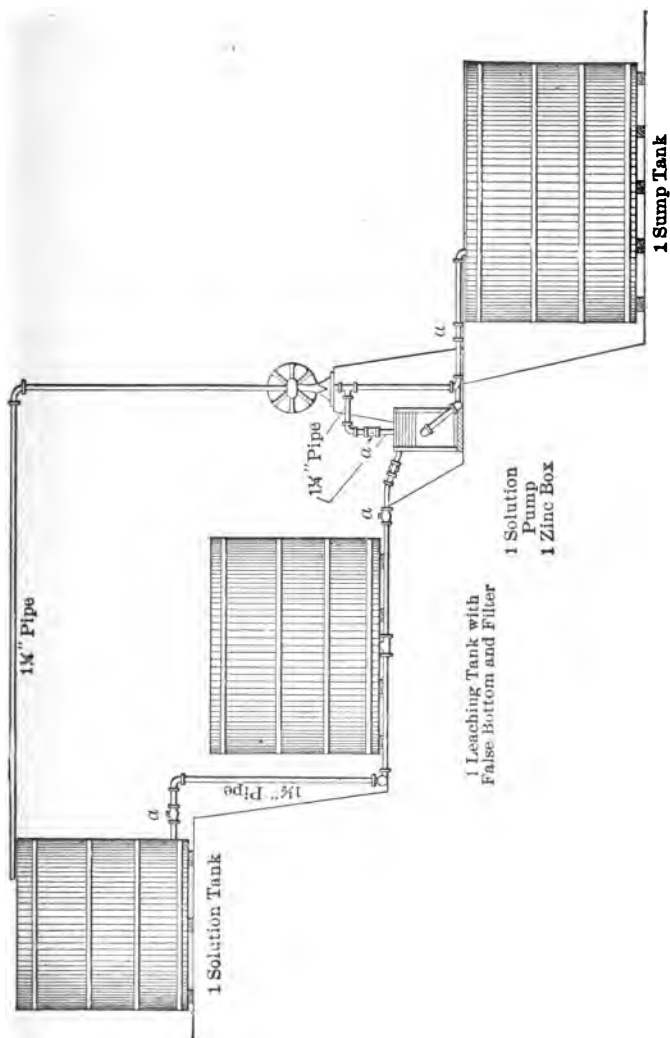


Fig. 5.—Three-ton Experimental Cyanide Plant.

Methods of operating the Cyanide Process.—The principal methods of operating the cyanide process are the *percolation method* and the *agitation method*, or some modification of these methods.

The Percolation Method.—In the percolation method the solution is allowed to percolate through the ore.

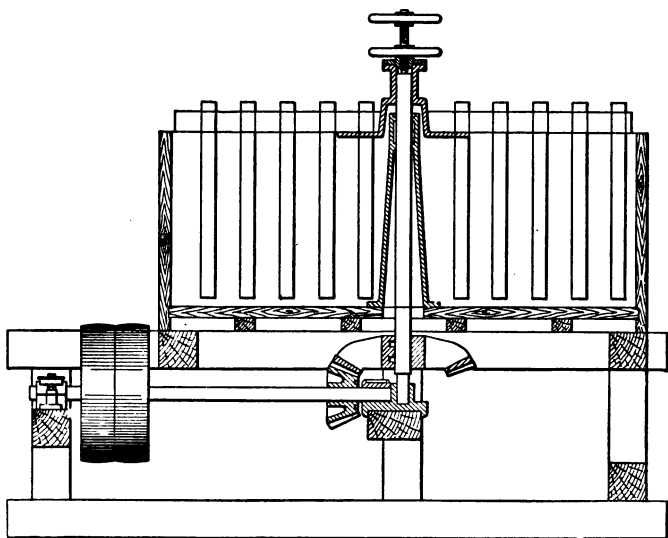


FIG. 6.—Agitator, Style A. When the extraction is complete the agitators are discharged into leaching vats, and filtration is accomplished by means of vacuum pumps, or the pulp is filter pressed.

This method has been described under “General Description of the Cyanide Process.”

The Agitation Method.—In the agitation method, after the cyanide solution is put on the ore, the ore is agitated by mechanical agitators (shown in Figs. 6

and 7), or by cold or hot compressed air, which is allowed to enter at the bottom of the tank (Pneumatic Process, Figs. 12 and 13). The air agitates the ore, and supplies oxygen. The use of oxygen in a cyanide solution is explained under "The Chemistry of the Process."

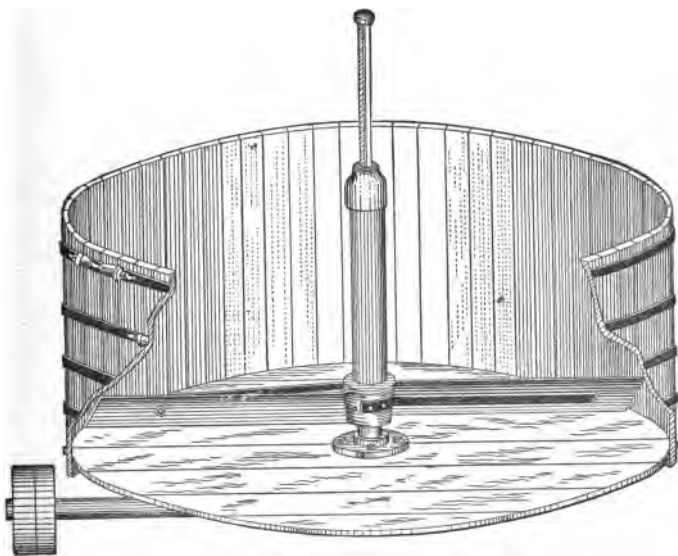


FIG. 7.—Agitator, Style B.

By the agitation method more gold is dissolved than by the percolation method; but power and additional potassium cyanide are consumed. Agitation may shorten the necessary time of contact of the solution with the ore sufficiently that there will be less consumption of cyanide by agitation than by percolation.

✂ **The Double-treatment Method** consists in treating the ore with a cyanide solution in a tank; and, after draining, the ore is transferred to another tank, and again treated with a stronger or weaker solution, which is sometimes followed by one or two weaker solutions.

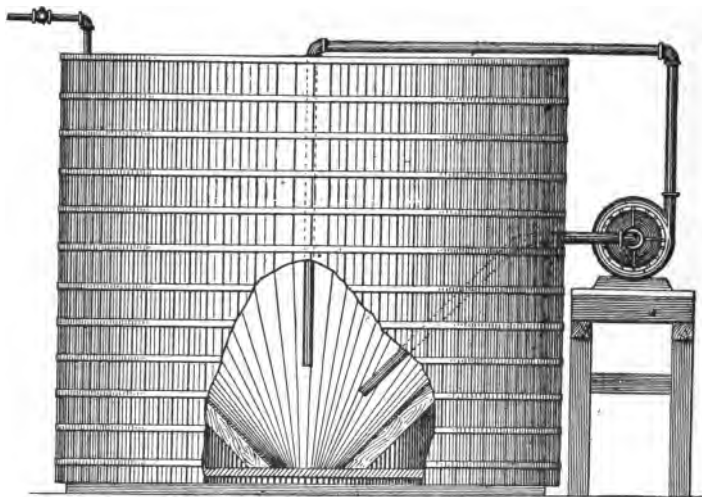


FIG. 8.—Agitation, by Centrifugal Pump.

This arrangement is used at many cyanide plants, particularly for the agitation of slimes in connection with decantation or filter-pressing. One advantage is that agitation can be readily started even if the material has settled compactly on the bottom of the tank, because, the suction-pipe being connected by a loose elbow, suction can be started on top of the charge, and as the material loosens up the weight of the pipe will cause it to gradually settle until finally the suction takes place at the bottom of the tank.

In the **Circulating Method** the solution is allowed to percolate through the ore, and is constantly pumped back on the ore—the ore being kept covered with the solution during the time of the treatment.

Sometimes, after the solution is put on the ore, the ore with the solution is pumped from one tank to another, which agitates the ore and supplies oxygen.

Methods of precipitating the Gold from Cyanide Solutions.—Precipitation of the gold from cyanide solutions is effected by zinc shavings (McArthur-Forest Process); by electricity (Siemens-Halske Process); by zinc shavings covered with lead (The Betty Process); by electricity on amalgamated copper plates covered with a layer of mercury (The Pelatan-Clerici Process); by zinc filaments; by sifting zinc dust into the gold-bearing cyanide solution, and keeping the solution with the zinc dust in agitation by compressed air; by charcoal. (Johnson)

CHAPTER II.

INTERFERING SUBSTANCES.

Acidity.—(If the ore contains an acid, decomposition of potassium cyanide results) (Sulphide ores or tailings, which have undergone oxidation, usually contain sulphuric acid.) (The soluble acid can be washed out of the ore by water, or the soluble and the insoluble acids can be neutralized by lime or soda.) (A large excess of lime or soda must be avoided, as it consumes zinc, if zinc is used to precipitate the gold; and, with ore containing a soluble sulphide, an alkaline sulphide would form, which interferes with the extraction.)

When sulphides of alkaline earths only are present, the lime may be added to the ore or to the solution; but when ferrous salts are present, an alkaline wash, followed by a water-wash, should precede the application of the cyanide solution (see under Iron).

Base Metals.—It has been generally stated that weak solutions of potassium cyanide attack the compounds of the base metals less in proportion to the gold than strong solutions. This has been attributed by McArthur and Forest to the "selective action" of potassium cyanide for gold. W. A. Dixon considers this action to be due to the larger proportion of oxygen

to cyanide in a weak solution, on the theory that cyanide attacks free gold so long as free oxygen is present, (then attacks the compounds of the base metals.) Julian and Smart * state that the proportion of gold and mineral matter of the ore dissolved in a weak solution is the same as in a strong solution: "the same proportionality exists with solutions of all strengths within working limits." For practical purposes it is sufficient to know that a weak solution of potassium cyanide gives better results than a strong solution.

(Base metals dissolve regardless of the strength of the solution, or the amount of oxygen it contains.) (Base metals are usually in the ore in the form of compounds, and gold in the metallic state.) (The compounds of the base metals exchange their bases for the potassium in the potassium cyanide, and form a compound with the cyanogen (metalepsy), or cause some other compound to be formed with the cyanogen.) Metallic gold being unable to replace the potassium in the potassium cyanide, it becomes necessary to add some element or substance, as oxygen, bromine, etc., which will set the cyanogen free, which will then dissolve the gold, and form a compound with it, or a gold-potassium cyanide.)

When a solution contains the required proportion of oxygen to dissolve the gold, the gold is more rapidly dissolved than when a solution contains less oxygen, which leaves the solution weaker in cyanide to be acted on by the base metals.

The action of potassium cyanide solution on com-

* "Cyaniding Gold and Silver Ores" by H. Forbes Julian and Edgar Smart.

pounds of the base metals depends somewhat on the physical condition and the chemical combination of these metals. (Compounds of zinc and copper physically hard are generally not much attacked by cyanide solution.) (Freshly precipitated oxides or hydrates of iron, zinc, copper, and lead, which may result from an alkaline treatment of the ore, are more readily dissolved than gold by a potassium cyanide solution.) The carbonates of the metals are also attacked by the cyanide solution.

Iron. (Iron in the ferrous condition is attacked by potassium cyanide solution, and thus consumes cyanide. Ferrous oxide or ferrous hydrate in the ore, or resulting from a lime or soda wash on ore containing ferrous compounds, should be oxidized to the ferric condition, by free access of air, before the cyanide solution is applied.)

(Basic ferrous salts resulting from oxidation of ore or tailings cannot be washed out by water, but they act on the potassium cyanide. By neutralizing these by lime or soda (see under Acidity), ferrous oxide and ferrous hydrate are formed, which should be oxidized to the ferric condition, by free access of air, before the cyanide solution is applied.)

Metallic Iron in a state of fine division consumes much cyanide. If the ore contains fine iron (some of which may be the result of crushing the ore in iron crushers and other iron machinery) or magnetic iron, separate such iron from the ore by magnetic separators, and, if it has sufficient values to pay to treat it, roast it to ferric oxide. It may need regrinding. It can then be treated separately or with the ore,

Soluble Sulphides.—Soluble sulphides decompose potassium cyanide, and form alkaline sulphides, which interfere with the extraction.) (The percentage of extraction falls with the accumulation of the alkaline sulphides in the solution. The sulphur in the alkaline sulphides can be removed from the solution by precipitation with lead salts, or changed to different compounds by oxidizers.

(Soluble sulphides should be turned into inert compounds before the cyanide solution is applied.) (Dead roasting before cyaniding is probably the only successful method so far applied.) Roasting to sulphates and washing the sulphates out by a preliminary water-wash has not proved successful, on account of the difficulty of turning all the sulphide into sulphates in this way.

McArthur and Ellis have covered by patent the use of suitable salts or compounds which, when added to the ore or solution, will form with the sulphur of the sulphide an inert sulphide. For this purpose, they give preference to the metallic compounds in the following order: Salts or compounds of lead, such as plumbates, carbonates, acetates, or sulphates of lead; sulphate or chloride of manganese, zincates, oxides, or chloride of mercury, ferric hydrate or oxide. The amount to be used must be ascertained for each ore by trials of a few samples.

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Copper.—Copper is attacked by cyanide solution, and thus consumes cyanide (see Copper, under Base Metals). If the gold is precipitated by zinc shavings, the copper precipitates on the zinc, forming a coating which prevents the precipitation of gold. An addition of

potassium cyanide solution to the gold-bearing solution, before it enters the zinc-boxes, or a coating of lead on the zinc by dipping the zinc shavings into a 1% or 2% solution of lead acetate, before they are put into the zinc-boxes, are proposed remedies.

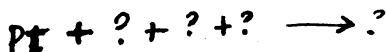
Cyanide solutions containing 0.3% or over of copper yield very poor results in extraction.* To remove most of the copper from the cyanide solution, by the lead-zinc couple, Superintendent Higgins of the Creston-Colorado plant, Minas Prietas, Sonora, Mexico, gives the following description, as practiced at that plant: "The solution coming from the tanks titrates about 0.23% KCN (potassium cyanide), and flows first through fourteen ordinary zinc launders, $18 \times 2.5 \times 2.5$ ft. in size, where the fine product is collected; secondly, from these launders the solution is taken through six other launders with zinc-lead in each compartment. I use 1.5 lbs. lead acetate to 15 lbs. zinc shavings; in other words, about 160 lbs. of lead acetate for 7500 tons ore." The lead-zinc couple is made by dissolving in distilled water, in each compartment of the zinc-boxes used for this purpose, 1.5 lbs. of lead acetate, after which 15 lbs. of zinc shavings are pressed into each compartment, and allowed to stand for half an hour or longer. The solution, after passing through the fourteen ordinary zinc-boxes, passes through the lead-zinc boxes, where a large per cent. of the copper is removed.

* W. H. Virgoe, *Institute of Mining and Metallurgy*, December, 1901.

(The removal of the copper from the ore by a preliminary treatment by sulphuric acid, followed by a water-wash, before the cyanide solution is applied, may prove satisfactory, where the value of the ore permits such treatment at a profit.)

✓ **Organic Matter.**—Organic matter precipitates gold, and extracts oxygen from the solution. Leaves, chips of wood, roots, and all other organic matter should be kept out of the water used, and out of the ore or tailings treated.

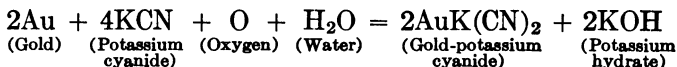
✓ Antimony, arsenic, iron, aluminum, magnesium, cobalt, manganese, zinc, interfere to a smaller or larger degree, according to the physical condition and chemical combination in which they are found.



CHAPTER III.

THE CHEMISTRY OF THE CYANIDE PROCESS.

It is generally accepted that oxygen is necessary to dissolve metallic gold by a solution of potassium cyanide, unless other substances are added that combine with the potassium and set the cyanogen * free. Elsner's equation is as follows:

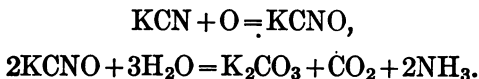


The equation for the solution of metallic silver is similar. There are other equations offered by different persons for the solution of gold, but all such equations contain oxygen. (For the purpose of dissolving a higher percentage of gold in less time, oxygen is sometimes supplied by the use of chemicals, or by mechanical devices.)

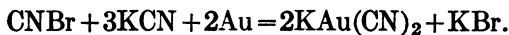
Among the chemicals that have been used are potassium permanganate, barium oxide, sodium peroxide, manganese dioxide, hydrogen peroxide, bleaching-pow-

* By cy-an'o-gen is meant the radical (CN).

der, etc. The oxygen may destroy some of the potassium cyanide:

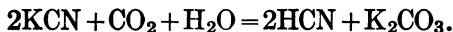


When chemicals are used that combine with the potassium and set the cyanogen free, no oxygen is needed to dissolve the gold. This may be illustrated by the use of cyanogen bromide:



Among the mechanical devices employed to supply oxygen are the forcing of compressed air through the solution and pulp (Pneumatic process), running the cyanide solution, before using, through a centrifugal pump which has air connection (Begeer process), and allowing the solution to run through open launders.

Decomposition of Potassium Cyanide.—This subject has been discussed to some extent under the subject of *Interfering Substances*. The mineral acids and many organic acids decompose potassium cyanide. Carbonic acid decomposes potassium cyanide:



Ore containing oxidized iron pyrites usually contains some sulphuric acid. With such ore the potassium cyanide solution, by several reactions and contact with the air, forms Prussian blue. This shows a large waste of cyanide. A mixture of ferrous and ferric sul-

phates with potassium cyanide also forms Prussian blue. This can be prevented by keeping the solution alkaline. This is known as *protective alkalinity*. Solutions work well on some ores with a protective alkalinity up to 0.3% or more. It should be borne in mind that, when soluble sulphides are in the ore, the alkali will form alkaline sulphides with the sulphur, which interfere with the extraction.

The action of the potassium cyanide on the compounds of zinc, aluminum, and other compounds has already been referred to under *Interfering Substances*.

Strength of Solutions.—Several experimenters have found that the rate of the solution of gold in potassium cyanide solutions increases from very weak solutions to solutions containing 0.25% potassium cyanide; after which the rate decreases. It has been found (by Maclaurin) that 0.01% potassium cyanide solution dissolves oxygen at about the same rate as ordinary water, and that this power increases up to 0.25% or 0.3%, after which it diminishes.

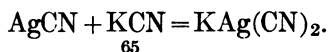
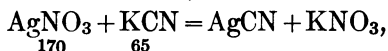
Weak solutions dissolve more gold per unit of potassium cyanide than strong solutions in the same time. The weakest solution that gives the best economical results, as shown by actual experiment with the ore, should be employed.

Reactions in the Zinc-boxes.—The precipitation of gold and silver by zinc was considered merely the substitution of the zinc for the gold and silver, but as free potassium cyanide must be present in the solution to effect the precipitation, other equations have been offered to explain the precipitation. As this subject

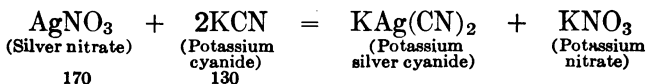
has not been settled, the equations have been omitted here.

Determining the Strength of Solutions.

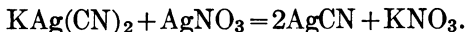
To determine the Strength of the Potassium Cyanide Solutions.—The determination of the potassium cyanide in a solution, by silver nitrate, is based on the fact that the silver cyanide, formed by the reaction of silver nitrate on potassium cyanide, is dissolved as long as free potassium cyanide is present, forming the double cyanide of potassium and silver:



Expressing the reactions by one equation,



When all the potassium cyanide is converted into a soluble double salt of potassium and silver cyanide, KAg(CN)_2 , any addition of silver nitrate will produce a permanent white precipitate of the simple silver cyanide:



If 17 grams silver nitrate are dissolved in distilled water and diluted to 1000 cubic centimeters (N/10

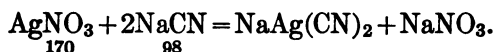


normal solution), in 1 cubic centimeter there is 0.017 gram silver nitrate; and each cubic centimeter silver nitrate corresponds to 0.013 gram potassium cyanide:

$$170:130::0.017:x. \quad x=0.013$$

By multiplying the number of cubic centimeters of silver nitrate consumed, before a permanent precipitate occurs, by 0.013, the weight of the potassium cyanide is found in the solution tested; from which the percentage strength * can be calculated.

✕ Some potassium cyanide on the market contains sodium cyanide. A sample containing sodium cyanide reported as potassium cyanide would show the solution to be stronger in cyanogen than it is. Sodium cyanide contains 53 per cent. cyanogen, and potassium cyanide only 40 per cent.:



$$170:98::0.017:X. \quad X=0.0098$$

One cubic centimeter of a deci-normal silver nitrate solution corresponds to 0.0098 gram sodium cyanide. By determining the percentage of sodium in the potassium cyanide, the per cent. of cyanogen can be calculated.

* By percentage strength is meant the amount in grams in 100 cubic centimeters. For example, by dissolving 1 gram pure potassium cyanide in water, and diluting the solution until the whole solution, containing the 1 gram potassium cyanide, measures 100 cubic centimeters, we have a 1 per cent. solution of potassium cyanide.

Methods to save Calculation.—If 13 cubic centimeters of the potassium cyanide solution to be tested are titrated with the deci-normal silver nitrate solution, the number of cubic centimeters of the silver nitrate solution consumed, divided by 10, gives the percentage of the potassium cyanide in the solution.

The silver nitrate solution can be made of any strength to save calculation. If 13.08 grams pure silver nitrate are dissolved in distilled water and diluted to 1000 cubic centimeters, and 10 cubic centimeters of the potassium cyanide solution are titrated by this silver nitrate solution, each cubic centimeter silver nitrate consumed corresponds to 0.1 per cent. potassium cyanide; or by dividing the number of cubic centimeters of silver nitrate consumed by 10, the percentage strength of the potassium cyanide is obtained, as above.

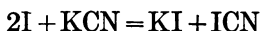
(The action on the potassium cyanide solution of the oxygen in the air, or the oxygen supplied by oxidizers, may result in the formation of ammonia.) As ammonia dissolves silver cyanide, and thus the titration would indicate too high results, it is best to add two or three drops of a 2% solution of potassium iodide to the sample of potassium cyanide solution before titrating it with the silver nitrate. The pale-yellow precipitate which forms, after all the potassium cyanide is consumed, is almost insoluble in ammonia. If much free alkali is present, the results may be too high.

To a potassium cyanide solution containing copper, two or three drops of a 2% solution of potassium iodide should be added before titrating it with the silver nitrate solution. Too much of the potassium iodide

affects the results when copper or much free alkali is present.

In some cyanide plants the sample of potassium cyanide is filtered through a little quicklime before it is titrated with the silver nitrate solution.

The strength of the potassium cyanide solution may be obtained by titrating it with a standard solution of iodine in potassium iodide, using starch solution as an indicator:



The iodine solution may be standardized by a sodium thiosulphate solution of known strength.

To bring a Weak Potassium Cyanide Solution up to Any Strength.—A strong solution of potassium cyanide is made by dissolving potassium cyanide in a separate tank, which solution is used to bring the working solution up to the required strength, after the latter has been in contact with the ore, and the gold has been separated from it.

For example, suppose 50,000 gallons of working solution of 0.25 per cent. strength, after having been in contact with the ore and the gold having been separated from it, has been reduced to 0.10 per cent. strength. How much of a 10 per cent. solution will it take to bring the 0.10 per cent. solution of 50,000 gallons up to 0.25 per cent. strength?

Let a = the strong solution, 10% strength;

b = the working solution after using, 0.10% strength;

c = the working solution before using, 0.25% strength;

d = the number of tons, lbs., gallons, etc. (in this example 50,000 gallons), working solution of 0.10% strength to be raised to 0.25% strength.

$\frac{c-b}{a-c} \times d$ = gallons 10 per cent. solution to be added to the 0.10 per cent. solution to bring it up to 0.25 per cent. strength.

$\frac{0.25-0.10}{10-0.25} \times 50,000 = 769.23$ gallons 10 per cent. solution to be added.

To reduce the Strength of a Solution.—Suppose we have 40,000 gallons of a solution of 0.24% strength, and we want to reduce it to 0.10% strength:

$0.10:0.24::40,000:X$. $X=96,000$ gallons.

Add water to the 40,000 gallons until the whole volume reaches 96,000 gallons, or add 56,000 gallons water ($96,000-40,000=56,000$) to the 40,000 gallons solution of 0.24% strength, and the strength will be reduced to 0.10% strength.

To make a Definite Volume of a Weaker Solution from a Stronger Solution.—Suppose it is desired to make 60,000 gallons solution of 0.20% strength from a solution of 10% strength:

$10:0.20::60,000:X$. $X=1200$ gallons.

Add water to 1200 gallons of the solution of 10% strength until the whole volume reaches 60,000 gallons, and the 60,000 gallons will have a 0.20% strength.

Danger in working the Cyanide Process.

Potassium cyanide is extremely poisonous. The building in which cyaniding is carried on, or in which cyanide solutions are standing, should be well ventilated. If this is not done, the men suffer from headache, faintness, and dizziness. It is recommended * that the following be prepared for cyanide poisoning:

- (1) 30 c.c. of 23 per cent. solution of ferrous sulphate.
- (2) 30 c.c. of 5 per cent. solution of caustic potash.
- (3) 2 grams of powdered magnesium oxide.
- (4) A metal receptacle of 1 pint capacity.
- (5) A stomach-tube.

Nos. 1 and 2 should be in hermetically sealed tubes, which can be broken into the receptacle and powdered magnesia and half a pint of water added, shaken up, and administered. This amount of antidote would account for 5 grams of cyanide of potassium, a quantity far in excess of what is likely to be drunk accidentally, but, to secure a sufficiently rapid reaction, the ferrous sulphate and alkali should be in considerable excess.

F. S. Tuttle recommends the swallowing of two drops of ammonia on a lump of loaf sugar; for external poisoning a warm bath containing washing-soda and common salt; and the inhalation of ammonia, when fumes are accidentally inhaled.

* By Dr. C. J. Martin and Mr. R. A. O'Brien, *Proceedings of the Society of Chemical Industry of Victoria*, Vol. I.

Methods for assaying Cyanide Solutions.

One cubic centimeter of cyanide solution weighs approximately 1 gram (1 cubic centimeter water at 4° C. weighs 1 gram). By dividing the number of cubic centimeters solution taken by the weight of 1 assay-ton (29.166 grams), the quotient will give approximately the number of assay-tons taken. This makes it easy to calculate the value to the ton of solution. Seven hundred cubic centimeters of solution are approximately 24 assay-tons ($700 \div 29.166 = 24$). When the percentage of gold in the solution is small, 1000 or 2000 cubic centimeters solution may be taken.

1. Take 1000 cubic centimeters solution, add excess of copper sulphate (the blue or green color of the filtrate indicates excess). Acidify with hydrochloric, nitric, or sulphuric acid, filter, wash precipitate, and scorify in the usual way; or assay by crucible method.

2. Put 500 cubic centimeters solution into a casserole, put under a hood with a good draft, acidify with nitric acid, boil for 15 minutes, then add $\frac{1}{2}$ gram silver dissolved in nitric acid, filter, and assay the precipitate and filter-paper as above.

3. Put 1000 cubic centimeters solution into a casserole or enameled iron dish, sprinkle 50 grams litharge into the solution, and evaporate to dryness on a sand-bath, water-bath, or iron plate. Scrape out the residue, mix with 7 grams potassium carbonate, 15 grams sodium bicarbonate, about 15 grams silica (powdered quartz or window-glass), 1 gram wheat flour, put into

a crucible, cover the charge with about 8 grams unfused powdered borax, and assay in the usual way.

The litharge must be sprinkled into the solution before evaporation begins.

4. To determine both Gold and Silver in a Solution.—Precipitate the silver with a solution of sodium sulphide, filter, dry, and assay. Precipitate the gold in the filtrate by zinc chloride, filter, etc., and assay.

5. Prepare a solution of mercuric chloride, and keep it in a glass-stoppered bottle. Take a measured quantity of gold-bearing solution, run in, from a burette, mercuric-chloride solution in excess; until no further precipitate is produced. Assay the precipitate.

6. Add 20 drops potassium bichromate to the measured gold-bearing solution. Run in strong silver nitrate solution until the deep-red color of chromate of silver appears. Add 100 grams zinc dust, and mix thoroughly. Dissolve the remaining zinc by sulphuric acid, filter, dry, etc., and assay.

A. F. Cross described 1 and 2 in *Jl. Chem. and Met. Soc. of S. Africa*, May, 1902. In "Gaze's Practical Cyanide Operations," 5 and 6 are described.

7. Pour 4 or more assay-tons of the solution to be assayed into a porcelain dish (casserole), add 10 c.c. of a 10% solution of acetate of lead, then 4 grams of zinc shavings, boil a minute, add 20 c.c. of hydrochloric acid. When the action has ceased, boil again, wash the spongy lead with distilled water, transfer it with a stirring-rod to a piece of filter-paper, squeeze into a compact lump, and place it into a hot cupel. The mouth of the muffle should contain a piece of dry pine

wood so that the muffle is filled with flame at the moment of introducing the spongy lead. (Alfred Chiddey.)

8. Take 3 to 10 assay-tons of the solution, according to richness, and bring to boiling. Acidify with hydrochloric or sulphuric acid to strong reaction with litmus paper. At end of 2 or 3 minutes, add 1 gram copper sulphate in solution, boil, add slight excess of either sodium or potassium sulphide to precipitate the copper. Continue boiling for a minute, or until evolution of hydrogen sulphide ceases. Filter through an eleven-centimeter filter. Remove the precipitate adhering to the sides of the casserole by a little cold water and rubbing. Fold filter, burn it in front of the muffle in a 2½-inch scorifier, add 20 grams test-lead, a small amount of borax, and scorify to 8 or 9 grams. (R. Stuart Browne.)

9. Draw off 100 to 500 c.c. of the cyanide solution. Place some sawdust or porous cotton in an iron pan or any convenient iron vessel. Pour the solution gradually upon the sawdust and allow it some time to be absorbed by the latter. Apply heat gradually to the iron vessel until all the solution is evaporated and the sawdust reduced to carbon. The residue, mixed with the proper fluxes, is transferred into a crucible, and then assayed in the ordinary way. Should the volume be too large, it can be quartered down. (Robert Grauer.)

10. Take 10 A. T. solution and heat it until hot; add ammoniacal copper nitrate until solution shows permanent blue color; add carefully excess of sulphuric acid and filter immediately; fold filter-paper and burn in a scorifier; transfer to a crucible, fuse and cupel. (Maurice Lineman.)

11. Sometimes it is desirable to know approximately what a solution carries, before one of the above tests can be made. Acidify with sulphuric acid, add excess of silver nitrate (until no longer a precipitate forms), filter and assay.

When the gold only is to be determined, it is best to add sufficient silver to the assay charge to insure separation of the button. When there are no foreign substances present, as sand, etc., wash the precipitate of gold or gold and silver to the tip of the filter, dust about 2 grams powdered lead over the inside of the wet filter, allow to drain, fold the filter to as small a form as possible, enclosing the precipitate with the folds, bring a red-hot cupel in front of the muffle, put 3 grams powdered lead into the cupel, place the folded filter-paper on the cupel, and allow the paper to burn at a low temperature in front of the muffle. Then cover the ashes with about 3 grams powdered lead, put the cupel into the muffle and cupel as usual. The silver for separation is added to the precipitate before folding the filter, when gold only is to be determined.

Testing Cyanide Solutions containing Zinc.*

1. **Total Cyanide.**—Add excess of sodium hydrate, 2 or 3 drops of a 2% potassium iodide solution, and titrate with silver nitrate solution. This gives the total cyanide (for reference below, we designate the

* L. W. Green, paper read before the *Institute of Mining and Metallurgy*, Oct. 17, 1901.

number of c.c. of silver nitrate solution consumed by T c.c.).

2. Protective Alkalinity. (This includes the alkaline hydrates, NaOH, KOH, the alkaline-earth hydrates, mainly $\text{Ca}(\text{OH})_2$, and half the monocarbonates in solution.)—Add excess of potassium ferrocyanide, twice the amount of silver nitrate consumed under 1, a few drops of phenolphthalein as indicator, and titrate with decinormal nitric acid. The amount of acid used will indicate the protective alkalinity (for reference as above, we designate this by *p*). The ferrocyanide precipitates the zinc, and the silver the cyanide. These precipitates may be filtered off before titrating with the acid.

3. Alkaline Hydrates.—Add excess of barium chloride (until no further precipitate forms). Then proceed as in the last test. (For reference, we designate this by *h*). Bicarbonates may be determined by adding a known quantity of standard alkali, and repeating the above test.

4. Total Cyanide, Ferrocyanide, Sulphocyanides, Chlorides, etc.—Add amount of acid (as shown by 2) to neutralize the alkali, a drop of potassium chromate, and then titrate with silver nitrate until a permanent faint-red color appears.

5. Ferrocyanides.—Add 10 c.c. decinormal sodium carbonate, the amount of silver nitrate consumed under 1, and shake well. All the ferrocyanide present will be precipitated by the zinc in the solution, and any excess of zinc will be thrown down as basic carbonate, and there will be an excess of sodium carbonate. Add phenolphthalein and neutralize slowly with decinormal

nitric acid. Add about 1 c.c. more of the nitric acid, shake, and add solution of sodium carbonate drop by drop till the clear solution is just faintly pink. Add excess potassium ferrocyanide. The reaction between the basic zinc carbonate and the potassium ferrocyanide makes the solution strongly alkaline. Titrate with decinormal nitric acid. This gives the amount of zinc less what has been precipitated by the ferrocyanide originally present (S).

6. **Zinc.**—Add sodium carbonate as in 5, and amount silver nitrate consumed in 4 to precipitate all cyanides, ferrocyanides, etc., which precipitates all the zinc as basic carbonate, and there will be an excess of sodium carbonate in the solution. Add phenolphthalein and neutralize as in 5. Add excess potassium ferrocyanide and titrate with decinormal acid. This gives the amount of zinc (also the copper and cadmium, if these are present) (Z).

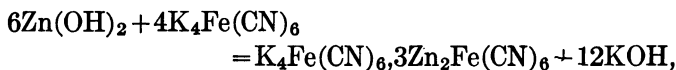
If in each of these tests 50 cubic centimeters of the cyanide solution were taken, decinormal nitric acid used, and the silver nitrate solution contained 13.05 grams silver nitrate in 1000 cubic centimeters solution, the following are the factors:

Total cyanide (as KCN)	$= T \times 0.02\%$
Protective alkali (as KOH)	$= p \times 0.0112\%$
Alkaline hydrate (as KOH)	$= h \times 0.0112\%$
Alkaline carbonates (as K_2CO_3)	$= (p - h) \times 0.0276\%$
Ferrocyanide (as $K_4Fe(CN)_6$)	$= (Z - S) \times 0.0351\%$
Zinc	$= Z \times 0.0081\%$

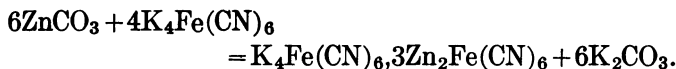
One cubic centimeter of decinormal potassium ferrocyanide precipitates 0.75 c.c. decinormal zinc solution from dilute neutral solutions. (Sufficiently exact for technical purposes.)

When a dilute neutral zinc solution is precipitated by excess of sodium carbonate, the excess of alkali being afterwards neutralized with decinormal acid, a precipitate of basic carbonate of almost constant composition is obtained: $3\text{Zn}(\text{OH})_2, 2\text{ZnCO}_3$.

Zinc hydrate or carbonate with excess of potassium ferrocyanide forms zinc ferrocyanide and potassium hydrate or carbonate:



or



These reactions are not complete until the alkali is neutralized.

Titration Complex Solutions.—On account of the impurities in solutions that have been used, the silver-nitrate test may not indicate the true amount of cyanide in solution. For such solutions J. E. Clennell gives the following method: "A measured volume of the solution is made strongly alkaline by the addition of caustic potash or soda. Sulphuretted hydrogen is passed into the liquid until it ceases to give a precipitate, avoiding a large excess, or, what is better, a concentrated solution

of pure sodium sulphide is added in slight excess. The solution is then well shaken and allowed to stand until the precipitate has subsided. A little lime may be added to assist the settling of the precipitate, in which case it can be filtered without difficulty. The clear filtrate is freed from excess of sulphide by agitating with litharge, which is best added in small quantities at a time, with constant agitation, until a drop of the liquid no longer gives the slightest black or brown coloration with a drop of lead acetate solution. A definite volume is then filtered off and tested with nitrate of silver in the ordinary way." (See also 1 above, under Testing Cyanide Solutions containing Zinc.)

CHAPTER IV.

DETERMINING THE APPLICABILITY OF THE CYANIDE PROCESS TO AN ORE.

LABORATORY tests are made to determine the applicability of the cyanide process to an ore. Laboratory tests, of course, are not conclusive; but, if they are properly conducted, they give an indication of what may be expected on a large scale. Before erecting a mill it is well to treat 50 to 100 lbs. ore by the best method as indicated by the laboratory experiments, which should be followed by treating from 3 to 6 tons.

(The percentage of extraction is usually higher, and the consumption of cyanide greater in laboratory tests than in a mill. It has been estimated that about one-third the amount of potassium cyanide consumed in laboratory tests is consumed in mill-runs, unless the consumption of cyanide is due to "cyanicides." By "cyanicides" is meant those substances that destroy the potassium cyanide, as free acid and other substances, some of which are mentioned under *Interfering Substances* (Chapter II).)

It is not unusual to get a higher percentage of extraction in a mill than in the laboratory.

A chemical analysis of the ore will give a good indication as to what preliminary treatment is necessary.

Extraction Tests.

1. The ore is crushed, sampled, and assayed.*
2. The ore is treated for interfering substances, such as acid, etc.
3. The percentage of extraction of the assay value is determined on samples of ore: (1) Crushed to different sizes; (2) in contact with the solution for different lengths of time; (3) for different strengths of solutions; (4) by different methods (percolation, agitation, etc.; roasted ore, dehydrated ore, etc.; by use of oxidizers, bromo-cyanogen, etc.).

After the ore is treated, the solution is filtered off and its strength determined, and the ore washed and assayed. The difference between the assay value before treatment and after treatment gives the extraction, or the amount of gold in the solution can be determined by one of the methods given under the *Assay of Solutions* (Chapter III).

Acidity.—Take 200 grams ore, cover with water, agitate for 10 minutes, filter, wash, and titrate with decinormal potassium hydrate solution (see Appendix). Litmus paper may be used as an indicator. This gives the *soluble* acidity.

Run an excess of decinormal potassium hydrate solution, from a burette, on the residue, agitate, filter, wash, and titrate back with decinormal acid solution.

* For the assay of ore for gold, silver, and lead, see the author's *Manual of Assaying*, published by John Wiley & Sons, New York.

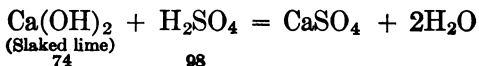
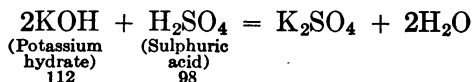
This gives the *insoluble* acidity. In both cases the ore must be washed until no more acid can be washed out, as shown by litmus indicator.

Total Acidity.—Take 200 grams ore, run an excess of decinormal potassium hydrate solution, from a burette, on the ore, cover the ore with water, agitate, filter, and wash as directed above. Add litmus solution to the filtrate and wash-water, and titrate with decinormal sulphuric acid solution.

Example.—Suppose 50 cubic centimeters decinormal potassium hydrate solution was run on the ore, and it took 8 cubic centimeters of the decinormal sulphuric acid solution to neutralize the filtrate and wash-water. Then it took 42 cubic centimeters of the decinormal potassium hydrate solution ($50 - 8 = 42$) to neutralize the acid in 200 grams ore.

One cubic centimeter decinormal potassium hydrate solution contains 0.0056 gram potassium hydrate. Having taken 200 grams ore, each cubic centimeter decinormal potassium hydrate solution corresponds to 0.056 lb. potassium hydrate to the ton (2000 lbs.) ore. ($0.0056 \div 200 = .000028$. $2000 \times .000028 = 0.056$.) (If 42 cubic centimeters potassium hydrate were consumed, it takes $.056 \times 42 = 2.35$ lbs.)

Assuming that the acid is sulphuric acid, how much slaked lime will neutralize the acid in the ore?



One cubic centimeter decinormal potassium hydrate solution corresponds to 1 cubic centimeter decinormal sulphuric acid solution; 1 part by weight potassium hydrate (in its power to neutralize sulphuric acid) corresponds to 0.66 part slaked lime ($74 \div 112 = 0.66$). Hence, in the example, where 42 cubic centimeters decinormal potassium hydrate solution were consumed to neutralize the acid in 200 grams ore, it takes 2.35 lbs. potassium hydrate, or 1.55 lbs. slaked lime, to the ton ore to neutralize the acidity of the ore ($0.056 \times 42 = 2.35$. $2.35 \times 0.66 = 1.55$).

To neutralize the Acid in Ore by Commercial Caustic Soda.—If commercial caustic soda is to be used to neutralize the acid in the ore, dissolve 10 grams of the soda to be used in water, and dilute to 1000 cubic centimeters. Take 200 grams ore, cover with water, add litmus solution, and run the soda solution, from a burette, into the mixture of ore and water. Stir the ore after each addition of solution. Add the solution slowly until a drop gives a permanent blue color to the litmus or litmus paper. Each cubic centimeter of the caustic soda consumed corresponds to 0.1 lb. of the same soda to the ton (2000 lbs.) ore. (In 1 cubic centimeter of the solution there is 0.01 gram soda. $0.01 \div 200 = 0.00005$. $2000 \times 0.00005 = 0.1$.)

Consumption of Cyanide.—Put 100 cubic centimeters of a 0.25% solution of potassium cyanide on 200 grams of ore. After the solution has been in contact with the ore for 24 hours (or whatever the time may be for the experiment), filter off 10 cubic centimeters of the potassium cyanide solution, and titrate

it with the decinormal silver nitrate solution (see Chapter III).

Example.—Suppose the potassium cyanide solution has been reduced to 0.10% strength. This shows a consumption of 1.5 lbs. potassium cyanide to the ton (2000 lbs.) ore. (200 grams ore were used, and only 100 cubic centimeters solution. $200 \div 100 = 2$. $0.25 - 0.10 = 0.15$. $0.15 \div 2 = 0.075\%$. $2000 \times .00075 = 1.5$.)

By running a *blank* with each experiment, that is, by allowing the same amount of solution and of the same strength to stand in the same kind of container, for the same length of time, under the same conditions (except not in contact with ore), etc., as that used for the experiment with the ore, the loss of cyanide not due to the ore may be found.

In making the tests indicated under the heading *Extraction Tests*, usually 200 grams ore are put into a glass-stoppered bottle with 100 cubic centimeters of potassium cyanide solution of 0.4% or 0.5% strength, and shaken for 20 minutes or longer. Ten cubic centimeters of the solution are filtered off and the strength determined. If the consumption of cyanide is high, the ore is treated for "cyanicides."

Subsequent experiments are carried on in percolators, fitted up as shown in Fig. 9. A filter is made from asbestos fiber, over which is placed a filter-paper, and on top of this the ore is placed; or an iron-wire gauze, supported on short iron-wire legs and covered with duck filter-cloth, may be fitted in the bottom. Larger samples can be treated in tubs, in barrels sawed in two, etc. These should be fitted with false bottoms (Figs.

21-25), covered with duck filter-cloth. When wooden vessels are used, they should be coated with paraffin, asphalt, or coal-tar paint.

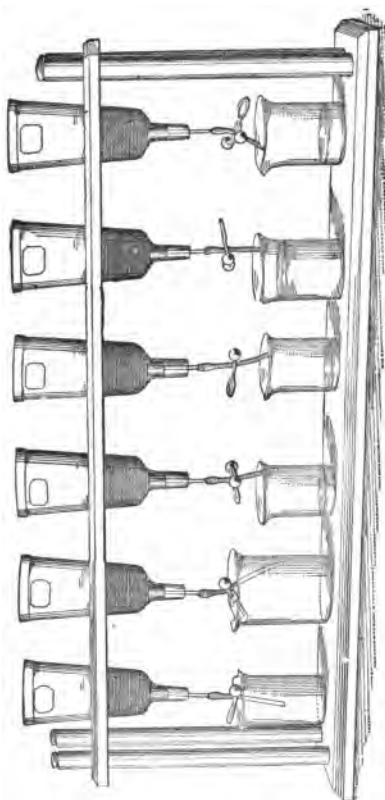


FIG. 9.

When ore is crushed to pass a 40-mesh screen or finer, it may not leach in the percolators. Fine ore can be treated in 6-inch ribbed funnels, Fig. 10.

Precipitation Tests.—The solution from the tests can be passed through glass tubes filled with zinc shavings; or if larger samples are treated, a small zinc

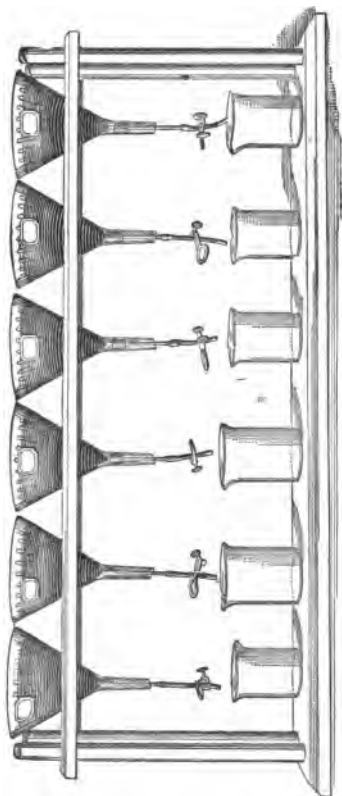


FIG. 10.

box may be used (Figs. 1 and 2). The amount of gold in a certain number of cubic centimeters of the solution is determined before the solution is passed through

the zinc shavings; and the amount of gold in the same number of cubic centimeters of solution is determined after the solution has passed through the zinc shavings, by one of the methods under *Methods for assaying Cyanide Solutions*. From these results the percentage of extraction by the zinc can be calculated.

Small portions of solution may be evaporated in dishes made from lead-foil, and the lead folded or rolled up and cupelled; but this usually gives too low results.

Coarse gold is usually amalgamated with mercury, as it takes too long to dissolve it with cyanide solution. Ore containing coarse gold is usually crushed by stamps and run over plates amalgamated with mercury; and, if the concentrate or tailing contains sufficient gold to pay for treatment, it may be treated by the cyanide process or the chlorination process.

Amalgamation tests can be made by shaking, in a bottle, for an hour or two, a pound of ore with an ounce of clean mercury and sufficient water to make a soft mud. Pan out the mercury; and save the tailing (and concentrate, if any), dry it, assay it, and, if it contains sufficient gold to pay for treatment, make cyanide tests on it as on an ore.

Amalgamation tests can also be made by panning the ore with mercury or by running the ore with water over an amalgamated plate.

CHAPTER V.

NOTES.

Strength of Solutions.—(In general, use the weakest solution that gives the best economical results.) The gold is precipitated from very weak solutions by electricity, zinc filaments, or the lead-zinc couple.

Temperature.—(The rate of the solution of gold increases with rise of temperature until a maximum is reached. The maximum point varies with solutions of different strengths. The rate of the solution of other metallic minerals also increases with the rise of temperature. Whether it is profitable or not to apply heat must be determined for each ore.)

Calcining.—Ores containing hydrous silicates often yield better results by giving them a preliminary calcination at a temperature not exceeding 300° F. If an ore contains lime or magnesia carbonate, and it is calcined at a much higher temperature, the ore will form a cement, and pack when the solution or water is applied.)

Crushing.—For porous ore coarse crushing is usually best; and for hard, dense ore fine crushing must be resorted to. At the Mercur mine, Utah, ore is crushed

to $\frac{1}{4}$ to $\frac{1}{2}$ inch; and at the Republic mine, Washington, ore is crushed to pass a 120-mesh screen. It should be noted that a laboratory crushing gives a coarser product than a mill crushing through the same size screen. Laboratory tests are usually begun on ore crushed to pass a 30-mesh screen.)

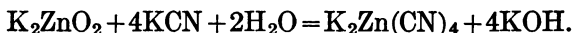
(**Roasting.**—When ore is roasted for the purpose of oxidizing the sulphur, arsenic, etc., the roasting should begin at a low temperature, the ore should be stirred, air admitted, and the temperature slowly raised, and finished at a dull-red heat. The ore should be roasted “dead,” or “sweet.” The roasting may produce soluble salts, which should be washed out of the ore before the cyanide solution is applied. The wash-water will contain some values, which can be recovered by precipitation.)

Difficulties in the precipitation by Zinc.—One of these difficulties is the appearance of a white incrustation said to consist of zinc ferrocyanide, which covers the zinc shavings and prevents further action. This does not appear when much free potassium cyanide or alkali is present. Formation of zinc ferrocyanide is increased by soluble salts of iron in the solution.

Used Solutions Less Powerful than Fresh Solutions of the Same Strength.—Alfred James found by experiment that the addition of lime to used solutions improved the extraction from free gold ores having a quartz gangue, but had the opposite effect on ore containing sulphur. In the latter case he found the addition of sodium sulphide (avoid excess), followed by the addition of a small amount of lead salt (acetate or chloride)

in excess, to be effective. After such additions, time must be allowed for precipitates to settle.

He also showed that the potassium-zinc oxide in the used solution combines with freshly added cyanide, which results in a weaker solution than the chemist had intended to make:

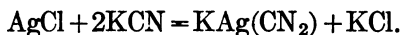


The accumulation of zinc in the solution is prevented by the action of the sulphides in the ore, or by the above-described treatment.

By the precipitation of gold from cyanide solution by zinc, a double cyanide of potassium and zinc, $\text{K}_2\text{Zn}(\text{CN})_4$, is formed. A solution of $\text{K}_2\text{Zn}(\text{CN})_4$ dissolves gold, but more satisfactory results are obtained by removing the zinc by the above treatment.

W. H. Davis has patented a process for "regeneration of the cyanide in the solution and clarifying of the latter" by "introducing into the solution an alkaline hydrate and then subjecting the mixture to the action of an alternating electric current."

Silver Ores.—As a general rule, potassium cyanide does not extract a very large percentage of silver from ore, under the same conditions that it extracts a large percentage of the gold. When the silver is in the ore as a chloride, bromide, or iodide, the extraction is high:



The reactions for the silver bromide and silver iodide are similar. No oxygen is needed to effect the solution of silver, when it is in the ore as a compound. When

the silver is combined with sulphur, arsenic, etc., the extraction is low, which is probably partly due to the formation of potassium sulphide:



When such ore is roasted with from 0.25% to 15% common salt, a silver chloride forms, which is soluble in potassium cyanide. Add the salt near the end of the roasting.

Low-grade ores containing both gold and silver may be profitably treated by this method. Ores running high in silver can be more profitably treated by other methods, as much potassium cyanide is consumed in the solution of silver chloride.

If roasted, the ore may need a preliminary water-wash to clean the ore of soluble salts. Some values will also wash out, which can be recovered by precipitating them.

Ore of the Dos Cabezas mines* of Sonora, Mexico, assaying 29 ounces silver and 0.1 ounce gold, by complete sliming, and treating for 24 hours by agitation by compressed air, the solution being warmed, the tailing assayed trace of silver and no gold. Consumption of potassium cyanide, 7.3 pounds per ton of ore treated.

The Clean-up.—At the end of two weeks or a month, the precipitates in the zinc-boxes are collected, and turned into bullion. These precipitates, in addition to the gold and silver, often contain lead, zinc, iron, lime, copper, etc.; and, even if they contain no copper, lead,

* M. B. Parker in the *Bulletin of the International Miners' Association*.

etc., they contain pieces of partly used up zinc. The coarse zinc is usually sieved off, and put back into the zinc-boxes. The liquid is pumped or siphoned off the gold slimes, and they are dried; or they are filter-pressed and then dried.

In cleaning up, the zinc shavings should be exposed to the air as little as possible, as they oxidize rapidly, after which they are almost useless for precipitation.

There are several methods by which these slimes are treated: (1) They may be melted with borax; (2) roasted, then melted; (3) treated with sulphuric acid, then melted; (4) treated with nitric acid, then melted. Alfred James found that (2), (3), (4) occasioned an additional loss to that sustained by melting with borax only, and that treatment with sulphuric acid, then melting, sustained the least loss among the three. His advice is to handle the gold slimes as little as possible, and to use lead-free zinc in the precipitation. The losses, in cleaning up, have been reduced by the use of a special filter-press.

Lead Smelting of Zinc-Gold Slimes.—P. S. Tavener * found that by fusing the precipitates with litharge, as in an assay, more can be recovered than by the roasting and acid treatment. By this process the zinc slimes are dried, roughly weighed to determine the amount of fluxes necessary, smelted in a reverberatory furnace with the fluxes and the gold-lead cupelled. The charge by weight is approximately as follows:

* *Journal of the Chemical and Metallurgical Society of South Africa*, October, 1902.

Zinc slimes.	100	parts
Litharge.	60	"
Assay slag.	10 to	15 "
Foul slag.	10 "	15 "
Silica.	5 "	10 "

Charge for fine zinc:

Fine zinc.	100	parts
Litharge.	150	"
Slag.	20	"

Sawdust is used as a reducing agent. One per cent. of the weight of the litharge of sawdust is mixed with the slime. If the proportion of litharge is in excess of 60 per cent., 1.5 to 2 per cent. of sawdust is required. No sawdust is added to the fine zinc. To determine the amount of slag necessary to produce a fusible slag, trials can be made in a crucible with a small quantity of slime or fine zinc. It takes about 30 per cent. less slag to make a fusible slag in the furnace than in the crucible. The resulting lead should not contain more than about 8 per cent. of gold, and 10 per cent. is the limit.

Additional Notes.

1. Lime is used to settle slimes.
2. Lime is preferred to soda for neutralizing acidity, as it does not cause formation of zinc ferrocyanide, which impairs the activity of zinc for precipitation.
3. If the solution is not clear, contains suspended matter, pass it through a sand filter before entering the zinc-boxes.

4. The formation of hydrogen gas in the zinc-boxes is caused by the action of potassium hydrate on the zinc. Excess of caustic soda or lime used in neutralizing the ore, if not washed out, will act on the zinc.

5. The solutions used on ores containing no sulphides are kept slightly alkaline. "Add soda or lime, a little at a time, till the solution flowing out from the extractor-boxes does not rise in cyanide test." (James.)

Excess of alkali is to be avoided with sulphide ores. "A little soda is necessary, and helps the zinc-box reaction, but use lime generally for neutralizing purposes, and keep your solutions so that they rise apparently in cyanide, if soda is added." ("Cyanide Practice by James.")

7. Sampling tailings in a vat by a glass tube or iron pipe may result in not getting a fair sample of the bottom layer.

8. From $\frac{1}{2}$ to 1 lb. cyanide is consumed per ton of ore treated. Some pyritic ores consume from 3 to 50 lbs.

9. From $\frac{1}{4}$ to $\frac{1}{2}$ lb. zinc is consumed per ton ore treated.

10. One lb. zinc should precipitate about 6 lbs. gold, but in practice it takes from 5 oz. to 1 lb. zinc to precipitate 1 oz. gold. Compounds in the solution, formed by the solution and the minerals in the ore, are, in a large measure, the cause of the high consumption of zinc.

11. Iron in contact with zinc causes solution of the zinc. The use of iron trays cannot be avoided.

12. Zinc shavings weigh about 5 lbs. per cubic foot in the boxes.

13. There should be three zinc-boxes in a cyanide works—one for the strong solution, one for weak solutions, and one for the washings.

14. Solutions rich in gold give a cleaner deposit on the zinc with less consumption of cyanide, the volume passing through the precipitation-boxes being less.

15. Most of the gold is precipitated in the upper compartments of the zinc-box. The zinc dissolves more rapidly in the upper compartments. As the precipitation proceeds, the zinc is transferred from the lower compartments to the upper ones, and fresh zinc is added at the foot of the box.

16. There are usually about ten compartments in a zinc-box, and the box has a fall of about $3\frac{1}{2}$ inches. The first and last compartments are usually left empty or supplied with sand filters to clarify the solution.

17. The surface of a charge of ore should be about 12 inches below the rim of the vat, and the solution about 3 inches above the ore.

18. Wooden vats should be coated with paraffin, asphalt, or coal-tar paint.

19. A stock or strong solution is not made until wanted, as it undergoes somewhat rapid decomposition.

20. Gold can not be precipitated by zinc shavings from a cyanide solution which does not contain at least 2 lbs. potassium cyanide to the ton solution. When the solution contains less than 0.05 per cent. KCN, the solution must be in contact with the zinc for an hour.

21. Zinc shavings used in the precipitation of gold from cyanide solutions will weigh about 6.5 to 7 lbs.

per cubic foot when well packed. It has been found advantageous to pack the precipitation boxes closely and firmly, as this offers a greater surface of zinc to the passing solutions than where they are loosely packed.

22. Foaming in the zinc boxes is due to excessive alkalinity.

23. Some commercial cyanide contains soluble sulphides, which should be removed by adding to the solution some slaked lime, and a requisite amount of lead salt (acetate or chloride). Filter the solution, or allow the precipitate to settle and decant the clear solution.

24. If a solution contains any reducing agents, these should be removed or oxidized before the solution is applied to the ore.

Useful Information.

It takes about 700 lbs. solution to saturate and cover 1 ton of ore.

It requires 100 gallons water to sluice 1 ton tailings out of a vat.

22 to 28 cubic feet = 1 ton dry raw ore.

20 to 26 cubic feet = 1 ton ore wetted down.

18 cubic feet of earth = 1 ton.

1 cubic foot of quartz in place = 165 lbs.

1 cubic foot of quartz broken = 94 lbs.

13 cubic feet quartz, unbroken = 1 ton.

1 cubic foot of water contains $7\frac{1}{2}$ gallons and weighs $62\frac{1}{2}$ lbs.

To find the capacity of a tank in cubic feet, square the diameter of the bottom in feet, multiply by 0.7854, and multiply the product by the inside height in feet.

CHAPTER VI.

SHORT DESCRIPTIONS OF SOME CYANIDE PROCESSES.

ALL the cyanide processes, with perhaps one or two exceptions, are covered by patent. The patent is on the method of operating or on the method of precipitating, or both.

The United States District Court for Idaho decided (in 1901) against the patent on zinc dust for precipitating gold-cyanide solutions, holding that the same had been anticipated by prior publications and patents. The case may be carried to a higher court for final decision.

McArthur-Forrest Process.—This process consists in applying dilute solutions of potassium cyanide to ore (not exceeding 8 parts of cyanogen to 1000 parts of water), and precipitating the gold from the solution by means of zinc shavings. The zinc shavings “are cut by a turning-tool from a series of zinc disks held between lathe-centers and turned.” The patent also covers the use of an alkali or alkaline earth to neutralize the acidity of the ore.

Siemens-Halske Process.—By this process the gold is precipitated from the solution by electricity. The anodes are iron plates, $7 \times 3 \times \frac{1}{8}$ in. thick, covered with canvas to prevent short circuits. The cathodes are

very thin lead sheets, stretched between two iron wires, and fastened (3 in each) in light wooden frames, 2×3 ft., which are suspended between the iron plates. The iron anodes cause the formation of Prussian blue by the reaction of ferrocyanide with iron oxide.

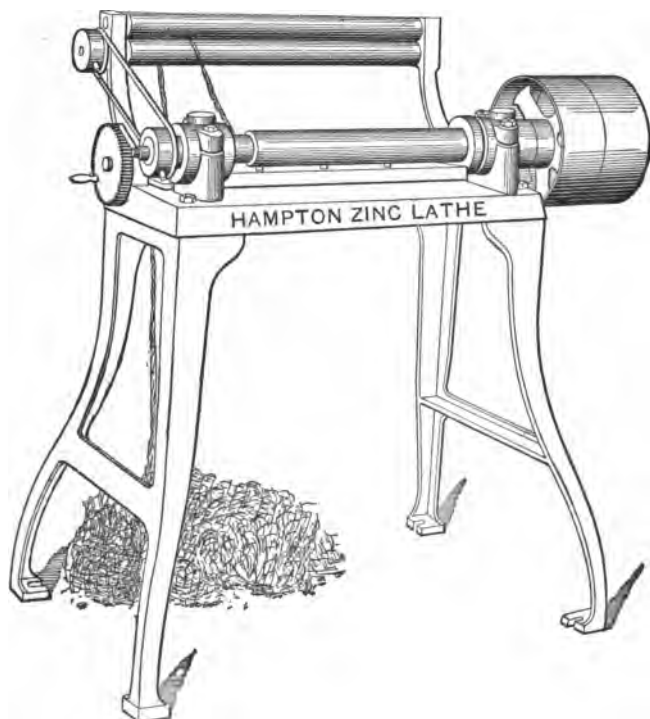


FIG. 11.—Hampton (Patent) Zinc Lathe.

Sometimes the space between the iron plates is filled with lead shavings, which take the place of the lead sheets, and offer a larger surface.

By this method of precipitation very weak solutions of potassium cyanide can be used to leach the ore. By employing very weak solutions of potassium cyanide, less cyanide is lost by "cyanicides," and in the solution that always remains in the ore; but it requires a longer contact of the solution with the ore.

The Pneumatic Cyanide Process.—After the cyanide solution has been put on the ore, compressed air is turned into the space beneath the false bottom in the vat, and is evenly distributed by means of a coil of perforated pipes. The air forces its way through every hole in the perforated false bottom, keeps the ore agitated, and supplies the oxygen for the rapid solution of the gold. It is claimed that nearly all sulphide ores, except those containing an excess of copper and antimony, can be profitably treated by this process. It admits of finer crushing, and requires less time of contact, than the ordinary percolation method. The slimes are forced, by the compressed air, to the surface, leaving the coarser and heavier portions of the ore on the filter, which facilitates the leaching.)

The Betty Cyanide Process.—This process effects the precipitation of gold from weak solutions. The zinc shavings are completely covered with a coat of lead by being moved about in a 10% solution of lead acetate. When the coating is complete, they are immediately transferred to the precipitation-tank, avoiding contact with the air. The gold is precipitated on the lead of this lead-zinc couple. This lead-zinc loses its precipitating power in the first compartment after a ten days' run, when it should be replaced by freshly prepared

zinc, and used to precipitate gold from solutions stronger in cyanide.

The precipitating-tank is 25 feet long, 6 feet wide,

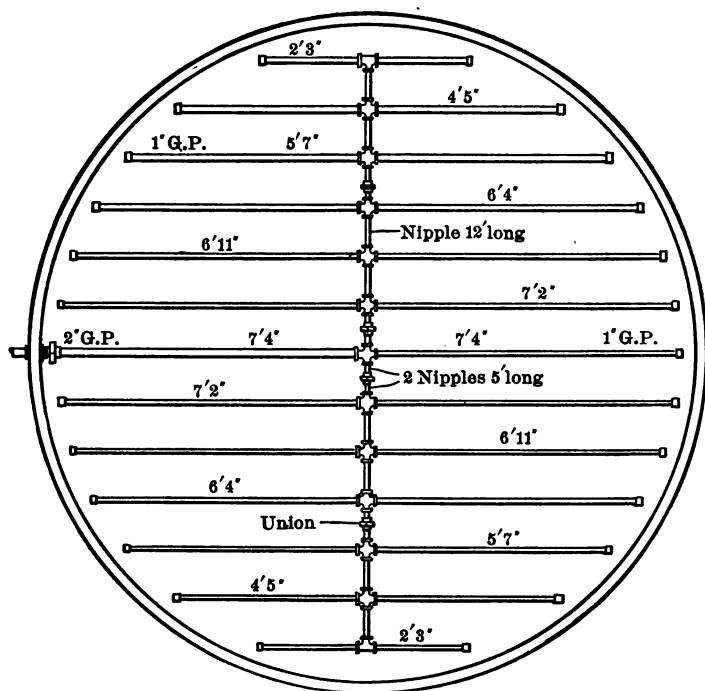


FIG. 12.—Arrangement of Pneumatic Air-pipe, arranged above false bottom of Leaching-tank. This illustrates the most simple pipe arrangement of the "Pneumatic" Process, but sometimes it is preferred to lead the main supply-pipe over the top of the Leaching-tanks, arrange the air-pipe in sections and connect same by different drop-pipes.

and 5 feet deep, divided into five compartments. Fourteen to fifteen tons of solution can be treated hourly. The solution enters the precipitating-tank at the top.

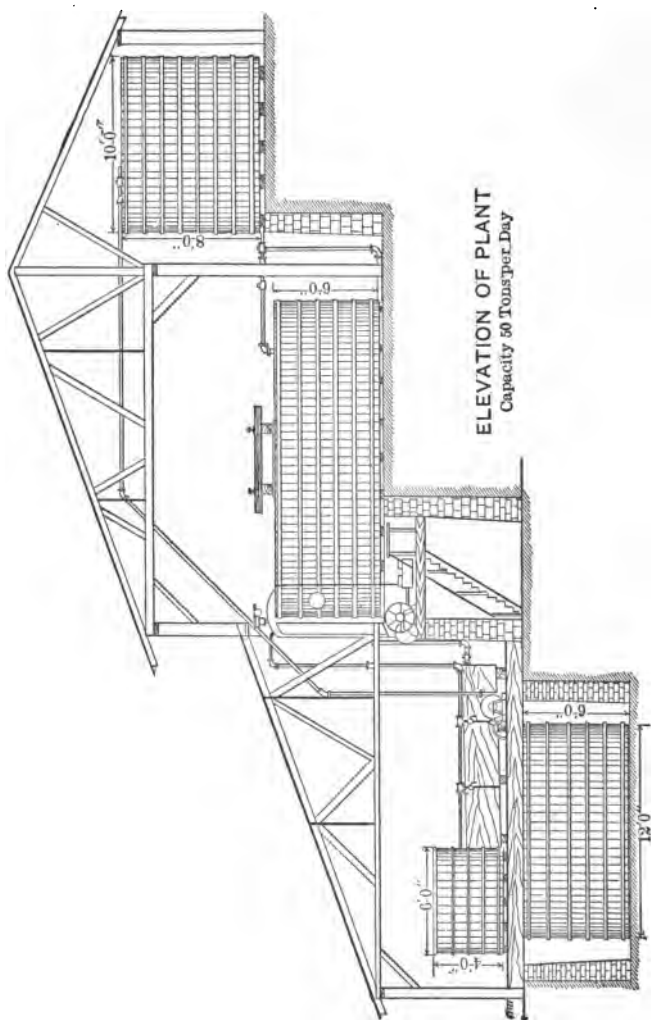


Fig. 13.—Elevation of 50-ton Plant.

With the gold-bearing solution, 75 gallons of a 2½% cyanide solution is allowed to run freely for four hours into the tank, bringing the solution up from 0.007% to 0.025%; 75 gallons of 1½% cyanide solution are now run in for 6 hours. About 12 hours after this, a small piece of cyanide is occasionally dropped into the gold-bearing solution, to keep the strength up to about 0.008 per cent.

The Godbe Agitation Process.—Generally, the finer an ore is pulverized, the higher the percentage of extraction. But fine ore packs, which interferes with the leaching, and a considerable amount of the gold-bearing solution cannot be washed out of the ore.

The difficulties of leaching slimes are overcome by introducing the cyanide solution, containing lime, below the false filter-bottom, agitating the ore by stirrers or compressed air, and displacing the solution by running the second solution in from the bottom, which is displaced in the same way by water. It takes about two displacements by the weaker solution, followed by a water-wash, to wash all the values out. Agitation increases the extraction.

The ore may be crushed wet, run into the vats, and the water displaced by the cyanide solution. By using a large amount of solution, a weaker solution of potassium cyanide can be used, and the proper strength of the solution can be kept in constant contact with the ore by continuous upward percolation.

The Begeer Cyanide Process.—This process differs from other processes in running the cyanide solution, before using, repeatedly through a centrifugal pump

which has air connections. In this way the solution is saturated with oxygen, and, it is claimed, will dissolve a higher percentage of gold and silver in a shorter time than a solution not so treated. The power required for this purpose is about one-third H.P. for 100-ton plant.

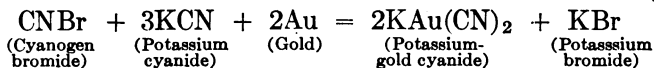
The Decantation Process.—Slimes are treated by this process. The slimes are agitated until the gold is dissolved, when they are allowed to settle, and the clear solution is drawn off from the top of the tank, and run through the precipitating-boxes. The slimes are washed several times with a repetition of agitation, settling, and drawing off of the clear solution. Sometimes the slimes are agitated by a centrifugal pump attached to the bottom of the tank (see Fig. 8).

Precipitation by Zinc Dust.—At the Golden Gate mill, Mercur, Utah, zinc dust is used to precipitate the gold from the cyanide solution. The zinc dust used is a blue bowder, the by-product from zinc smelting. Thirty tons of solution is pumped into a precipitating-tank; and, while the tank is filling, the precipitate from former precipitations, which contains unconsumed zinc dust, is stirred up by introducing air into the bottom of the tank through a pipe, at 10 to 15 lbs. pressure. Five lbs. fresh zinc dust is used for 30 tons solution. Beginning when the tank is half full, the zinc dust is sieved into the solution, at intervals, until all the zinc is added. The sediment is then thoroughly stirred up by moving the air-pipe about the bottom, after which the pipe is removed, and the precipitate allowed to settle for half an hour. The liquid is then drawn off through an opening in the side of the tank, about 8

inches above the bottom, and passed through the filter-presses. The precipitation is rapid. About $1\frac{1}{2}$ lbs. zinc dust is consumed per ounce of gold recovered.

At the Republic mill, Washington, twenty tons of solution are precipitated at each charge by sifting in the necessary quantity of zinc dust, while the solution is being agitated by means of compressed air, introduced through a pipe into the bottom of the tank. About 1.25 lbs. zinc dust is consumed per ounce gold precipitated, or 0.80 lb. per ounce combined gold and silver.

The Bromo-Cyanogen Process.—This process consists in using a mixture of cyanogen bromide and potassium cyanide. It is the Sulman-Teed process, known in some places as the "Diehl":

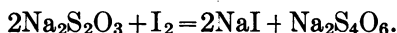
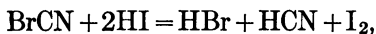
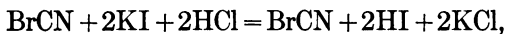


It will be noticed that by this process no oxygen is necessary to dissolve the gold.

At Deloro, Canada, the mixture of potassium cyanide and cyanogen bromide solution is run on the concentrates to the amount of about one-third of the weight of the ore. The circulating method is used. The solution percolates through the ore, is brought up to strength and kept in continued circulation for 24 hours, then the ore is drained, turned over by shoveling, and the circulation continued until assays show the extraction complete. The ore is then given a wash by a weak cyanide solution, followed by a water-wash. Consumption per ton of concentrates: Potassium cyanide, 2 lbs.;

cyanogen bromide, 0.5 lb.; zinc dust (precipitation), 0.19 lb.; treatment, 80 to 100 hours; extraction, 87% to 94%.

The strength of the potassium cyanide is found by the silver nitrate test. To find the amount of cyanogen bromide, acidify by hydrochloric acid, add slight excess of potassium iodide, starch solution as indicator, and titrate the liberated iodine by decinormal sodium thiosulphate. One cubic centimeter decinormal thiosulphate corresponds to 0.0052 gram cyanogen bromide:



Excess of bromo-cyanogen should be avoided, as it is no longer operative after passing the zinc boxes. The amount of cyanogen bromide should not be greater than 25% of the potassium cyanide present in the regular solution.

The Holderman Method of extracting Gold by Cyanide.

—The main feature of this method is the Holderman patented filter-tank, having its sides and ends, as well as its bottom, covered with filter material, stretched over A-shaped slats, giving a percolating and leaching area equal to the whole surface of the interior of the tank. The bottom of the tank slopes from back to front, allowing an easy discharge of treated ore through several front gates. The tanks are about 5×7×12 feet in dimensions. About three of these tanks are arranged in stair-like succession, which constitutes a plant for the treatment of ore. The uppermost tank is provided

with an agitator. In this tank the ore is treated with cyanide solution for from 2 to 6 hours. At the end of this time the solution is drawn off through the filter, the agitator being kept in motion, and the ore is then washed into the next lower tank with a weak cyanide solution, and allowed to stand a certain length of time, then the solution is drawn off through the filter, and the ore washed into the next lower tank.

The Moore Process.—This process employs a peculiar hollow truncated cone revolving on a horizontal axis in which the ore is treated with cyanide solution, agitated, aerated, and the sand is separated from the slime. The slime with the solution flows down inclined troughs into agitation-tanks. Agitation is accomplished by a centrifugal sand-pump, and the slime is finally filter-pressed.

The Hendryx Process.—This process is designed for the rapid extraction of gold, silver, and other metals from their natural ores.

The Process.—The ore is first crushed in a weak chemical solution containing one-fiftieth of one per cent., or less, of cyanide of potassium, and the pulp passed over amalgamated copper plates; then the ore-pulp solution is agitated in the Hendryx Agitator, in which the gold and silver from the ore pulp is dissolved and precipitated by either electricity or zinc.

The solutions are now removed and returned to a subsidiary tank on their way to the storage-tank, hereinafter designated as "battery storage," from which the crushing and grinding machinery is supplied, an additional set of plates for electrical deposition is installed. (Fig. 15.)

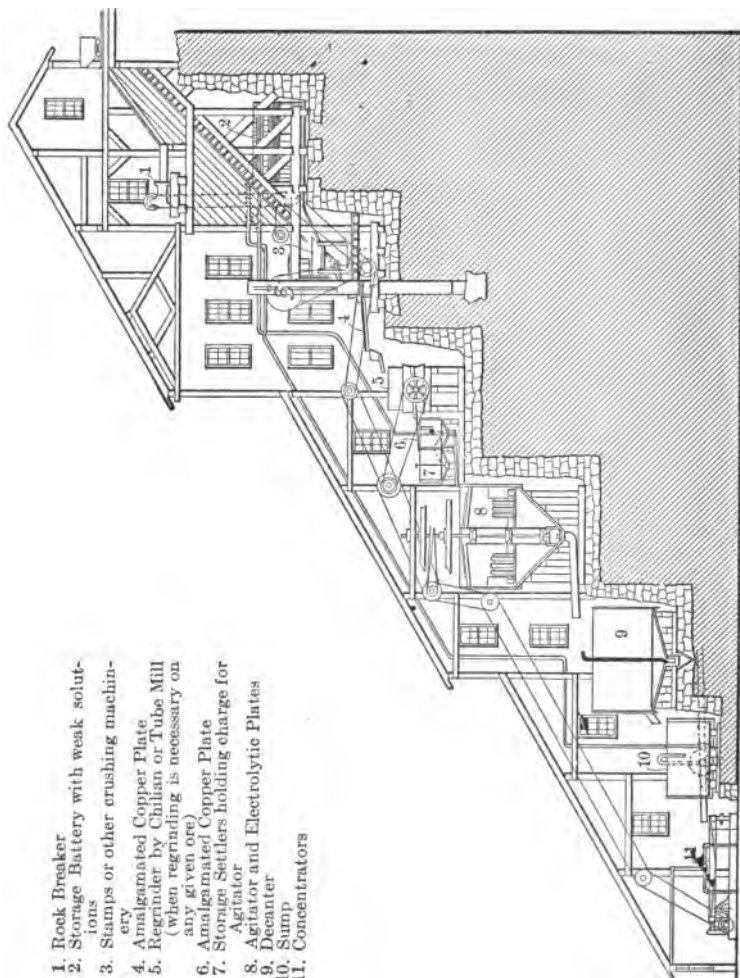


Fig. 14.—The Hendryx Process.

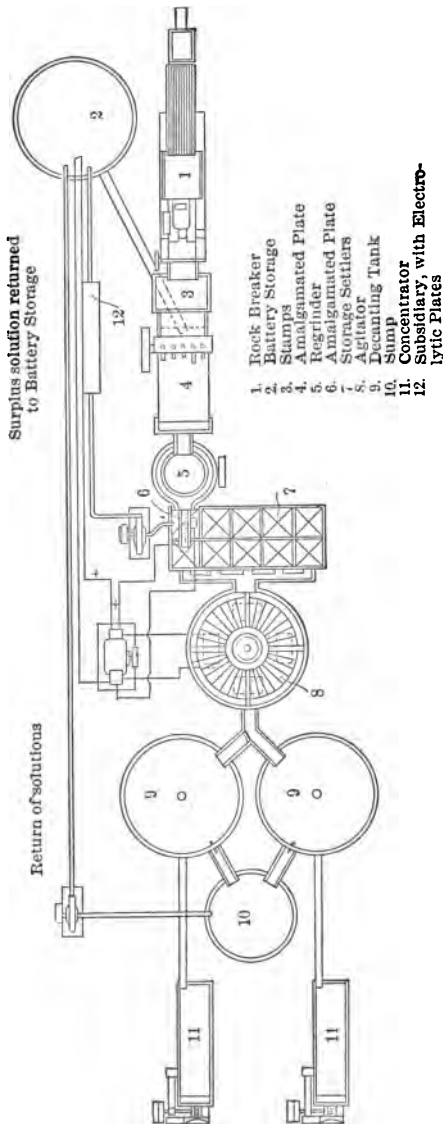


Fig. 15.—The Hendryx Process.

In the event of there being values left in the pulp, owing to insoluble metallic compounds or alloys, or owing to the lack of having ground the ore sufficiently fine to permit of the solutions coming in contact with the metals, then a further step in the process is carried out, consisting of concentration (tables being shown in Figs. 14 and 15).

The Hendryx Agitator (see Fig. 16) is designed for the rapid extraction of gold and silver from unseparated sands and slimes by means of agitation and aeration in weak chemical solutions containing cyanide of potassium or sodium. Simultaneously the values are deposited on plates by electricity, cyanide is regenerated, and the fouling of the solutions is prevented.

Essential Mechanical Feature.—The essential mechanical feature of the Agitator (Fig. 16) consists of a cylindrical tank having a conical bottom. In the center of the tank is a circular well which extends nearly to the top and bottom, supported by braces from the side of the tank and having a circular apron at the top which slopes gradually toward the circumference and permits the ore-pulp solution, in flowing over it, to be spread out into a thin sheet and to absorb an abundance of free oxygen. In this well is a hollow shaft carrying a driving-pulley near the top and a number of screw propellers or pulp-solution lifting-wings. The discharge-valve is connected to a screw for opening and closing. Outside the well and between the apron and the bottom of the tank, the anode and cathode frames or the electrolytic filtering-envelope are supported from the side of the tank and hold the anodes

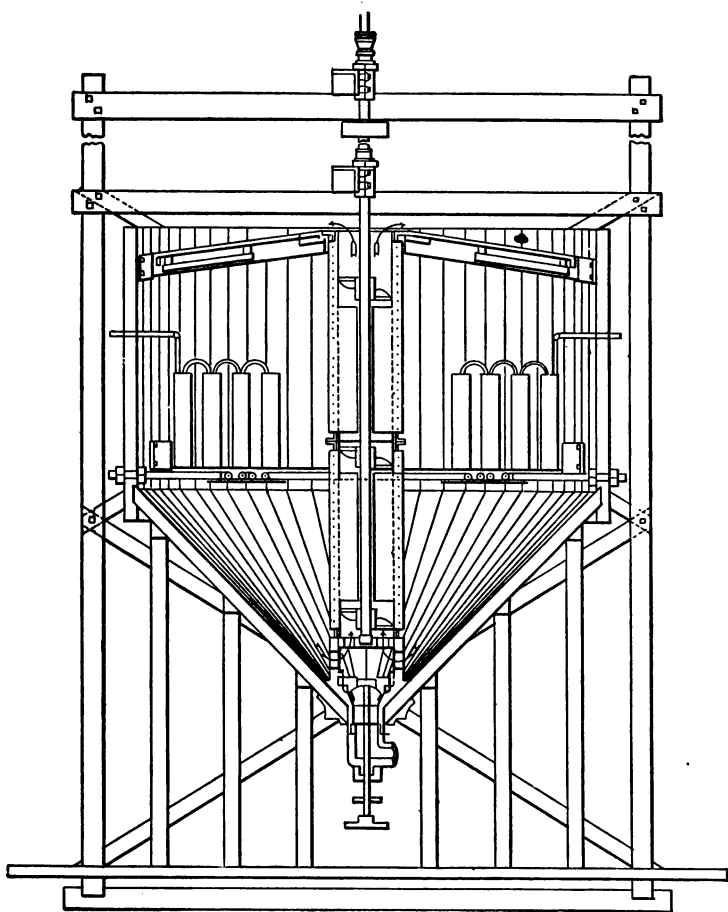


FIG. 16.—The Hendryx Agitator.

and cathodes which are supplied with electrical current. A coil of pipe serves for raising the temperature of the charge by means of steam or hot water passing through it. The revolutions of the propellers in the well produce a strong upward current, thus resulting in a rapid and uniform circulation of the pulp upward to the aerating apron, where it spreads out into a thin sheet, losing its velocity and gently falling from the edge of the apron into the great mass of ore-pulp solution, where its velocity is again reduced, and then gently dropping down through the electrically charged plates or envelopes.

Cannot scour Plates.—As the violence or force exerted by this method of agitation is within the tube or well, and as the plates are outside the well, no scouring of the plates is possible, thereby removing all danger of scouring the deposited metals from off the plates, which is so common in all of the old arm or paddle stirring devices.

Agitator cannot be clogged.—The charge in this agitator is homogeneous and cannot be clogged. A charge of 76 tons of pulp (30 tons ore, dry weight) in a 16-foot agitator was settled 48 hours on account of the breaking of a countershaft. Upon starting up, the pump was throwing its full capacity in three minutes, and the charge was in perfect agitation in ten minutes.

Power required.—The power required to operate a machine of 80 to 100 tons of ore pulp, containing one or two tons of solution to one of ore, is about eight horse-power, and the whole contents of the tank are brought up and over the aerating or distributing plate once in five or ten minutes, at the pleasure of the operator. by an increased or decreased speed of the pump.

Sample Specifications and Prices of Cyanide Plants for 48 Hours' Contact.

SPECIFICATIONS.

- 2 Solution-tanks.
- 4 Leaching-tanks, including false bottom, style A or B, Duck Filters and Bottom Discharge-doors.
- 2 Gold-solution Tanks.
- 3 Zinc Precipitation-boxes (2 for strong solution and 1 for weak solution).
- 2 Sump-tanks.
- 1 Solution-pump.
- Acid-proof Paint in sufficient quantity to coat all tanks on the inside.
- Tank-connections.
- Cyanide Stop-cocks.
- And all necessary Pipe and Pipe Fittings.

Plants up to and including 25-ton capacity are furnished with 100 pounds of Zinc Shavings, and larger plants with a Zinc Lathe.

(These specifications do not include crushing machinery, furnaces, etc.)

Price.

Daily Capacity at 25 Cubic Feet per Ton.	Approximate Weight.	Cost.
10 tons	16,780 lbs.	\$1,000 00
15 "	18,760 "	1,140 00
20 "	22,290 "	1,335 00
25 "	26,430 "	1,460 00
40 "	43,655 "	2,155 00
50 "	50,570 "	2,535 00
60 "	57,150 "	2,720 00
75 "	63,260 "	3,040 00
100 "	77,485 "	3,800 00
125 "	101,320 "	4,300 00
150 "	113,000 "	4,940 00
200 "	134,150 "	6,080 00

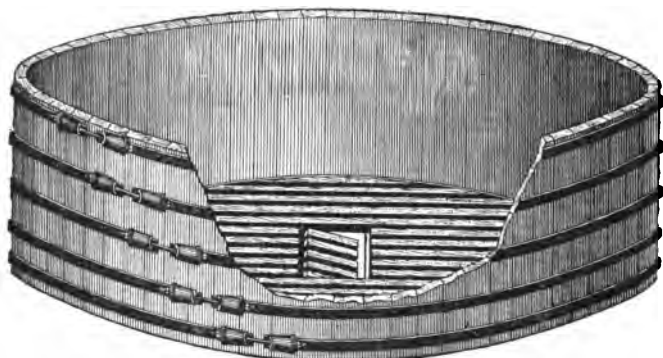


FIG. 17.—Leaching-tanks

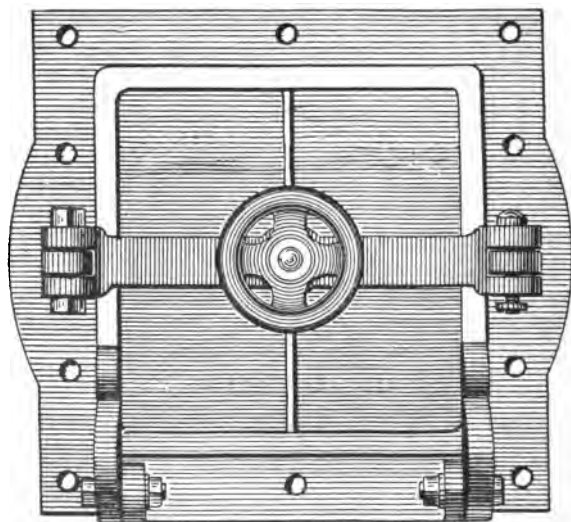


FIG. 18.—Bottom Discharge-doors. Size of opening 10×10 in., or 16×16 in. These sluice-doors consist of heavy cast-iron frames with iron doors attached by bolt hinges. They are kept water-tight by means of rubber gaskets placed around the edge of the door proper. Bottom discharge-plugs are used where it is not desirable to open doors from below a tank. These plugs are removed from inside by means of a handle extending to top of tank.



Size.	Weight.
8×13 in.....	115 lbs.
8× 8 "	90 "



FIG. 19.—Side Discharge-doors.

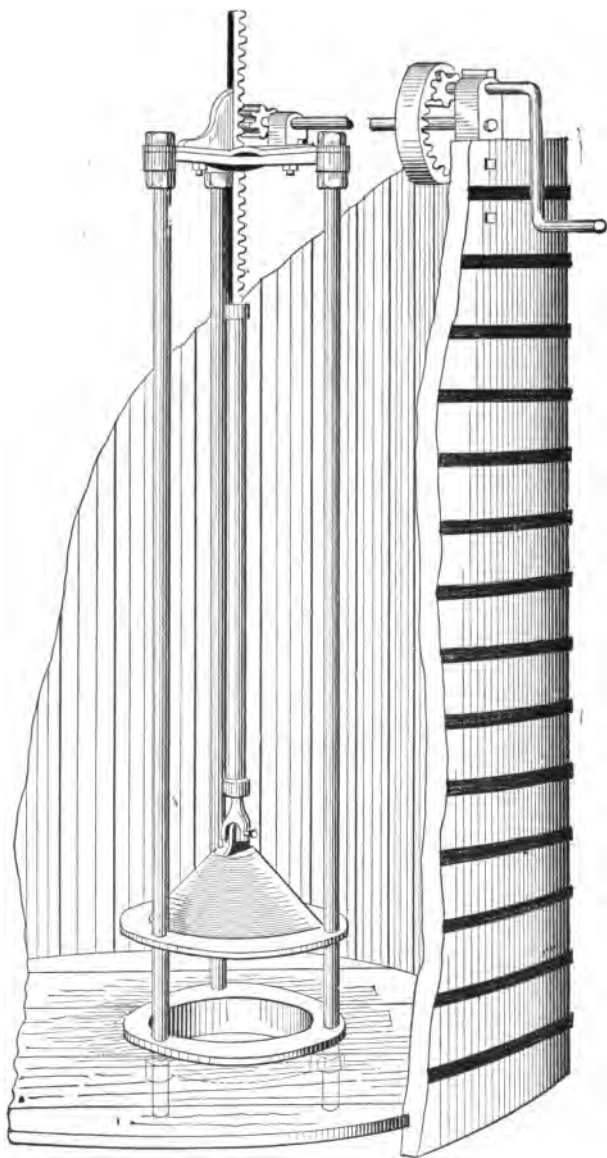


FIG. 20.—Bottom Discharge-gate.

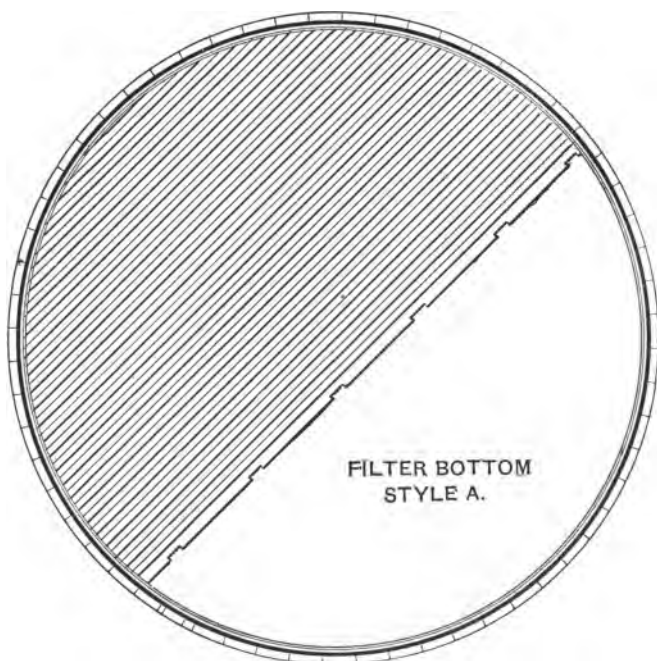


FIG. 21.—False Bottom for Leaching-tanks. This bottom consists of a grating built of Oregon-pine strips laid about $1\frac{1}{4}$ inches apart, and for the protection of the filter a thin band is placed around the outer edge of these strips. Each strip is crozed to allow of free flow of solution.

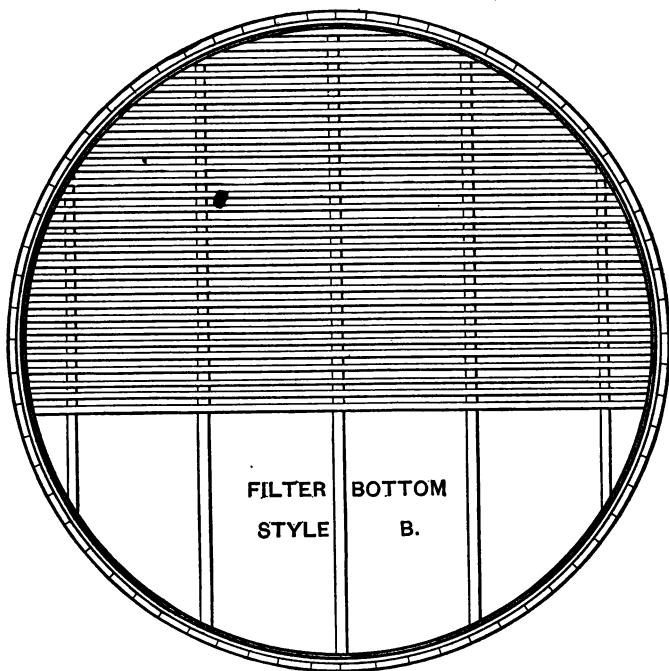


FIG. 22.—False Bottom for Leaching-tank. The bottom slats of this filter bottom are similar to style "A," but placed farther apart on which a grating is placed, consisting of 1-inch Oregon-pine strips, laid 1 inch apart, with a suitable strip around the outer edge of slats to prevent filter-cloth from being cut.

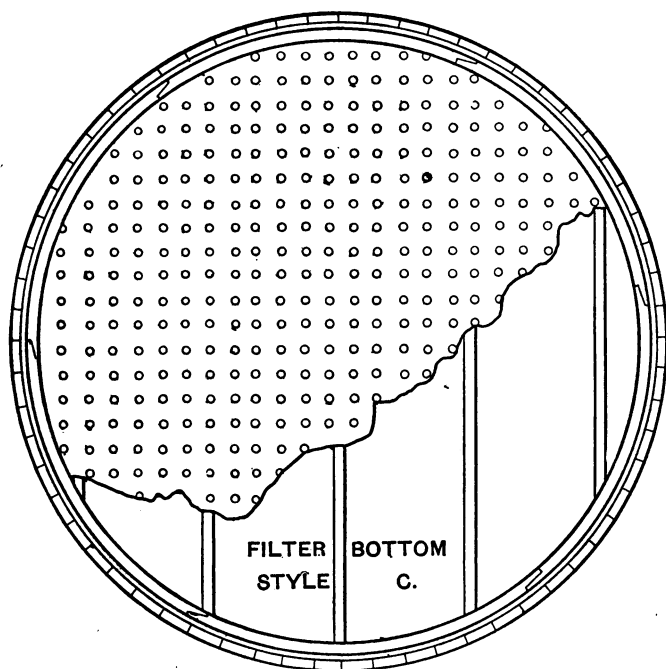


FIG. 23.—This filter is constructed of 3×4-inch segments, forming a complete circle; within this circle are placed the sill-pieces which support a 1-inch Oregon-pine floor provided with holes bored at equal distances.

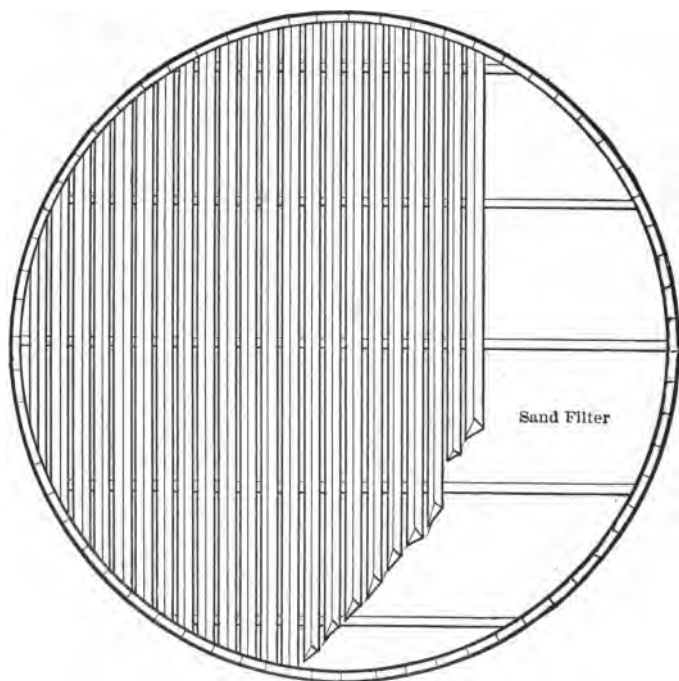


FIG. 24.—Sand Filters. In some localities this filter is very much in use. It consists of triangular slats resting on regular sill-pieces. The spaces between the slats are filled with gravel and sand, level with the upper edge, and no filter-cloth is used in connection with them.

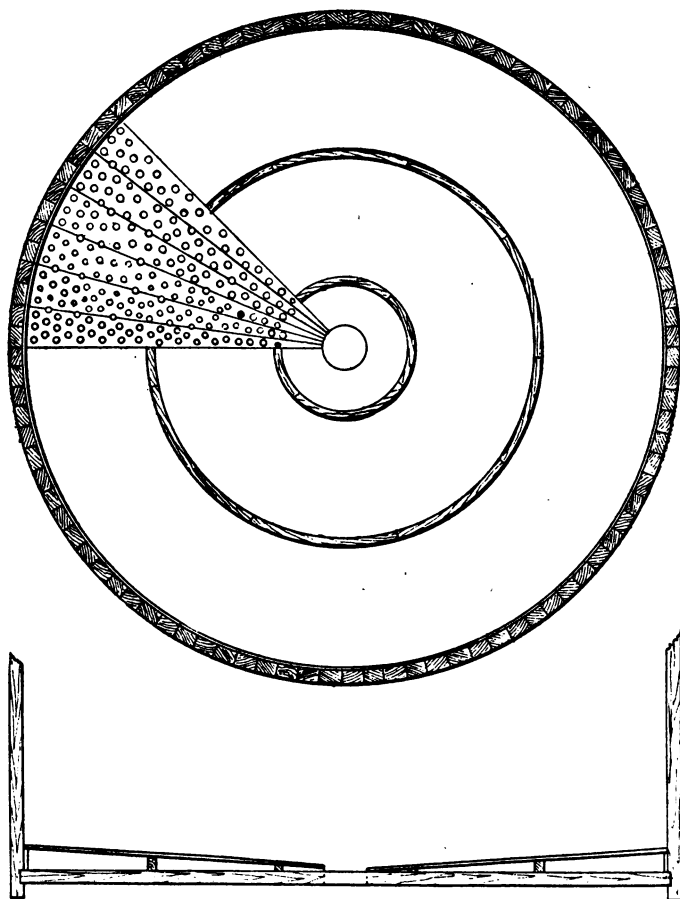


FIG. 25.—False Bottom, Style D, sloping 5° to center.

This Filter Bottom consists of 1-inch Oregon pine, perforated with $\frac{1}{2}$ -inch holes, 1 inch center to center.

The bottom rests upon circles 3 ft. apart. The circles are built up to different heights, with openings crozed in lower part for free flow of solution.

Tailings Discharge is placed in center of tank, but tanks above 36 ft. diameter have special arrangement for four bottom doors.



FIG. 26.—Fairfax Exploration Co.'s Cyanide Plant.



FIG. 27.—Cyanide Works, "Myall United" Gold-mine,
McPhail, N. S. W.

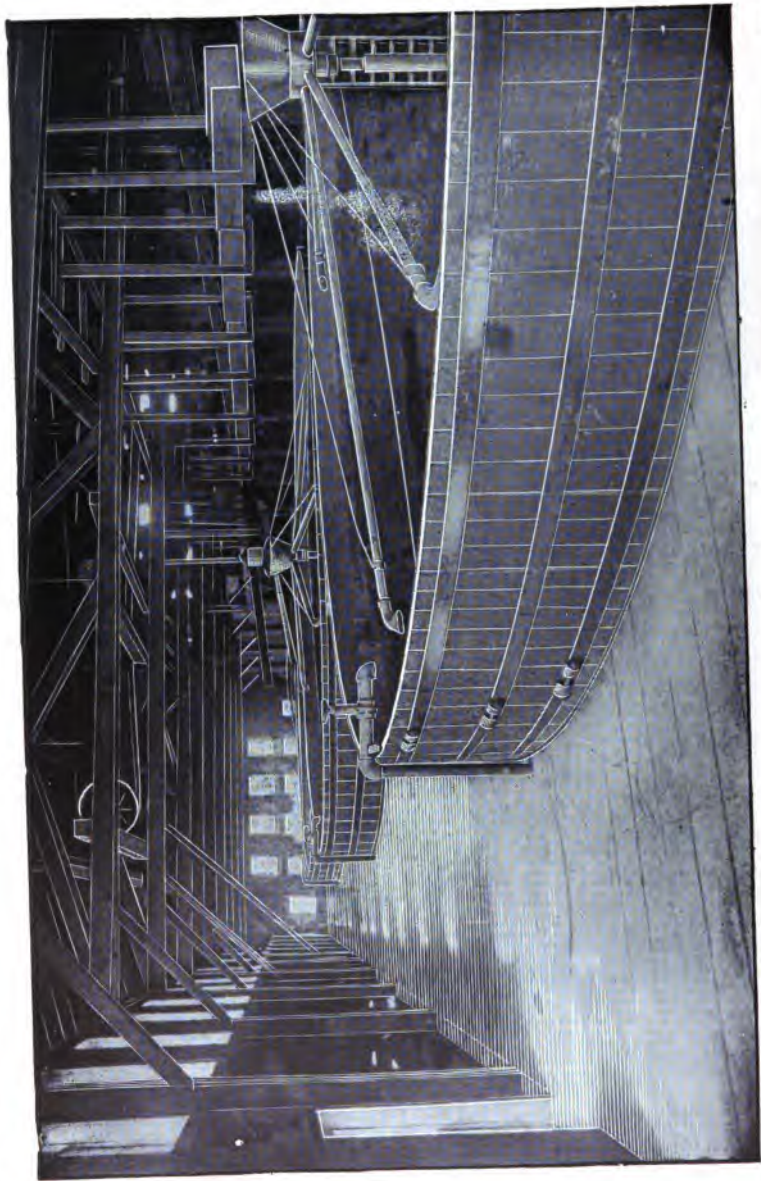


FIG. 28.—Leaching-tanks with Pulp-distributors.
Cyanide Plant, erected by Liberty Bell Gold Mining Co., Telluride, Colo.



FIG. 29.—Clean-up Room at the Cyanide Plant of Liberty Bell Mine, showing Iron Vacuum-tanks, Zinc Precipitating-boxes, Vacuum-pumps, Solution-pumps, Acid-tanks.

APPENDIX.

Volumetric Analysis.

By volumetric analysis the quantity of a substance in a solution is determined by another solution of known strength, with which the solution to be determined reacts. When the reaction is complete, a precipitate forms, or ceases to form; a color appears, or disappears; a change of color occurs, or a change of color is produced in an *indicator* put into the solution for this purpose.

End Reaction.—The point in the operation where the reaction is complete, as indicated by change of color, etc., is called the *end reaction*.

Titration is the operation of running a standard solution into the solution to be determined, and observing the end reaction: the operation of volumetric analysis.

Normal Factor.—The amount in grams of the reagent in 1 cubic centimeter of a normal solution is called the *normal factor*.

Indicators.

Litmus.—Grind litmus in a porcelain mortar with a little alcohol. Transfer to a flask or beaker, and pour some boiling alcohol on the litmus. After standing for some time, pour off the alcoholic solution, which is of no value. Pour distilled water on the litmus, let stand for some time, and then filter the solution. Add dilute hydrochloric acid, drop by drop, to the blue solution until it turns violet (not red). Keep the solution in a loosely covered vessel. Alkalies turn the solution blue, and acids red. Free carbonic acid, CO_2 , interferes with the production of the blue color: it must be boiled out of the solution. It cannot be used by gaslight.

Methyl Orange.—Dissolve 1 gram in 1000 cubic centimeters of distilled water. It is colorless, or faint yellow with excess of alkalies, and red, or pink, with acids. It is not affected by carbonic acid or sulphuretted hydrogen. It cannot be used in organic acids or hot solutions.

Phenacetolin.—Dissolve in alcohol, in proportion of 2 grams per 1000 cubic centimeters of alcohol. It is used in titrating potassium hydrate, sodium hydrate, or calcium oxide in the presence of their respective carbonates. Alkalies turn it pink, and acids yellow.

Starch Solutions.—Mix 1 gram starch with a little cold water, then pour 100 cubic centimeters boiling water on the starch. Mix and filter. The solution should be cold when used. It must be made frequently, as it will not keep for a long time. If a little

salicylic acid or pure common salt is added, the solution will keep for a long time. It is used in titrating with iodine. Iodine colors starch blue.

Phenolphthalein.—Dissolve 1 gram in a liter of 50% alcohol. It is used in titrating organic acids, and fixed caustic alkalies in presence of carbonates. It gives no color with bicarbonates, and cannot be used for the titration of free ammonia and its compounds, or for the fixed alkalies, when salts of ammonia are present. It is purple in alkaline solutions, and colorless in acids.

Standard Solutions.

Standard Solutions.—When a solution of definite strength of a chemical is made, by means of which the strength of other solutions is determined, it is a standard solution. The strength of which to make a standard solution depends on the peculiar reaction such a solution has with the solution to be tested.

Write the equation for the reaction between the standard solution and the solution to be titrated, from which the value of the standard solution to the solution to be determined will be seen.

Normal Solutions contain the hydrogen equivalent of the reacting element in grams in 1 liter (1000 cubic centimeters, at 16° C. or 60° F.). A normal solution is indicated by the letter N. Solutions of less strength are indicated as N/10 (1/10 normal, or decinormal), etc. If a substance is univalent (example, HCl), the full molecular weight is taken. If bivalent (example,

H_2SO_4), half the molecular weight is taken, etc. This rule does not hold good in all cases. (Read paragraph above beginning, Write the equation for the reaction. . .)

All standard solutions should be made with the greatest care and accuracy, and should be kept in well-stoppered bottles. Before using a solution, the bottle containing the same should be well shaken, to take up the liquid that may have evaporated and condensed on the sides of the bottle, and to mix the solution thoroughly. Burettes and all other measuring-vessels must be clean and dry before a standard solution is measured by them.

Vessels in which weighed or determined samples for standard solutions are dissolved must be thoroughly rinsed into the measuring-cylinder before the solution is diluted to the determined volume.

Bottles with glass stoppers and burettes with glass stop-cocks should not be used for caustic-alkali solutions, as they are liable to stick fast.

To make a Decinormal Potassium Hydrate, KOH, Solution.—Potassium hydrate cannot be accurately weighed. The solution must be standardized against an acid which can be accurately weighed.

Weigh out exactly 6.300 grams perfectly pure crystals of oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O} = 126$; $126 \div 2 = 63$; $63 \div 10 = 6.300$), dissolve in distilled water, and dilute to exactly 1 liter (1000 cubic centimeters*). This is a decinormal oxalic acid solution. Each cubic centimeter contains 0.0063 gram of the acid.

* Cubic centimeters is usually abbreviated to c.c.

Weigh out, approximately, 5.600 grams pure potassium hydrate ($\text{KOH}=56$; $56 \div 10 = 5.600$), dissolve in distilled water, and dilute to about 800 cubic centimeters.

Take 10 cubic centimeters of the potassium hydrate solution, to which add several drops of litmus solution (or methyl orange), and titrate from a burette by the decinormal oxalic acid solution, until the litmus (or the methyl orange, if used) turns red. Note the number of cubic centimeters of oxalic acid consumed. Make a duplicate titration. Do the same with 20 cubic centimeters of the potassium hydrate solution. If the results agree closely, take the average.

Example.—Suppose it took 12 cubic centimeters of the oxalic acid solution to neutralize 10 cubic centimeters of the potassium hydrate solution. The potassium hydrate solution must be diluted until 1 cubic centimeter of the oxalic acid solution will exactly neutralize 1 cubic centimeter of the potassium hydrate solution.

Suppose there are 740 cubic centimeters of the potassium hydrate solution remaining after the above trials:

$$10:12::740:x. \quad x=888.$$

Add distilled water to the 740 cubic centimeters of potassium hydrate solution, until the whole volume reaches 888 cubic centimeters; or add 148 cubic centimeters water to the 740 cubic centimeters potassium hydrate solution ($888 - 740 = 148$).

Now titrate again. One cubic centimeter of the acid should exactly neutralize 1 cubic centimeter of the potassium hydrate solution. Each cubic centimeter contains 0.0056 gram potassium hydrate.

To make a Decinormal Sulphuric Acid, H_2SO_4 , Solution.—Take 2.8 cubic centimeters strong sulphuric acid (1.84 sp. gr.), and pour it into about 800 cubic centimeters distilled water (pour the acid into the water, not the water into the acid). After the mixture is cool, take 10 cubic centimeters of the acid and titrate from a burette with the decinormal potassium hydrate solution (use litmus or methyl orange as an indicator). Note the number of cubic centimeters of potassium hydrate consumed to neutralize the acid. Make a duplicate titration. Do the same with 20 cubic centimeters. If the results agree closely, take the average.

Example.—Suppose it took 12 cubic centimeters of the decinormal potassium hydrate solution to neutralize 10 cubic centimeters of the sulphuric-acid solution, and suppose there are 768 cubic centimeters of the sulphuric acid solution remaining, after the above trials:

$$10:12::768:x. \quad x=921.6.$$

Add distilled water to the 768 cubic centimeters sulphuric acid until the whole volume reaches 921.6 cubic centimeters, or add 153.6 cubic centimeters water to the 768 cubic centimeters sulphuric acid ($921.6 - 768 = 153.6$). After the mixture is cool, titrate again. One cubic centimeter decinormal potassium

hydrate solution should exactly neutralize 1 cubic centimeter of the sulphuric acid solution. Each cubic centimeter contains 0.0049 gram sulphuric acid.

To make a Decinormal Nitric Acid, HNO_3 , Solution.—Take pure nitric acid and standardize it against decinormal potassium hydrate solution, in the same way as directed for sulphuric acid. In 1 cubic centimeter decinormal nitric acid solution there is 0.0063 gram of the acid.

To make a Decinormal Iodine Solution.—Procure three watch-glasses (accurately fitting on one another) of convenient size. Weigh two glasses accurately. Take about 12 grams iodine, and mix it with about one-fourth its weight potassium iodide on the third glass. Cover this glass with one of the weighed glasses, which should not touch the mixture. Apply heat to the glass containing the mixture. After the iodine has sublimed on the upper glass, remove it and cover it with the other weighed glass, cool, and weigh.

Example:

	Grams.
Weight of glasses and iodine.....	32.000
Weight of glasses only.....	20.000
Iodine.....	12.000

A decinormal iodine solution contains 12.685 grams iodine per 1000 cubic centimeters.

$$12.685:12::1000:x. \quad x=945.99.$$

After weighing the glasses containing the iodine, place them immediately in a beaker containing a solu-

tion of nearly twice as much potassium iodide as there is iodine by weight (the potassium iodide must give no blue color to starch, when dilute sulphuric acid is added to a solution of the potassium iodide used for this purpose).

Now transfer the solution to a graduated cylinder, and fill it up to the required volume, 945.99 cubic centimeters. Each cubic centimeter contains 0.012685 gram iodine.

Second Method.—Put some pure iodine in a weighed, glass-stoppered, weighing-bottle, and weigh the bottle and iodine (the bottle must be kept tightly stoppered, as iodine is very volatile).

Make a solution of nearly twice as much potassium iodide by weight as iodine taken, as in the first method. With this solution wash the iodine out of the weighing-bottle into a graduated cylinder. Be careful that all the iodine is transferred to the graduated cylinder.

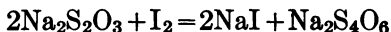
Example.—Suppose the iodine (less the weight of the bottle) weighed 6.000 grams.

$$12.685:6::1000:x. \quad x=472.99.$$

Add distilled water to the cylinder until the whole volume reaches 472.99 cubic centimeters. This is a decinormal iodine solution, the same as under the first method.

Third Method.—Powder pure crystals of sodium thiosulphate, dry by pressing between folds of blotting-paper, and weigh out exactly 24.8 grams ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O} = 248$. $248 \div 10 = 24.8$). Dissolve in distilled water

and dilute to exactly 1000 cubic centimeters. This is a decinormal solution of sodium thiosulphate. Each cubic centimeter contains 0.0248 gram sodium thiosulphate. As only half the sodium reacts with iodine, the whole molecular weight of the sodium thiosulphate will be contained in a liter of a normal solution:



Dissolve iodine in distilled water containing potassium iodide, as directed in the first method. Have the iodine solution stronger in iodine than decinormal (by not diluting to that point). Draw 10 cubic centimeters of the sodium thiosulphate solution from a burette, add a little starch solution, and run in, from a burette, the iodine solution. As soon as the iodine is in excess, it blues the starch solution. Make a duplicate, etc., as directed in other examples.

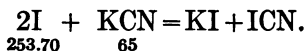
Suppose it took 8 cubic centimeters of the iodine solution to neutralize 10 cubic centimeters of the sodium thiosulphate solution, and there are 500 cubic centimeters of the iodine solution remaining:

$$8:10::500:x. \quad x=625.$$

Add distilled water to the iodine solution until the whole volume reaches 625 cubic centimeters, or add 125 cubic centimeters water to the 500 cubic centimeters iodine solution ($625-500=125$). This is a decinormal solution of iodine, the same as under the first and the second method.

Iodine solutions should be occasionally tested, as they change after standing for some time. In a well-stoppered bottle wrapped in brown paper to exclude the light, the solution remains unchanged for a long time.

To determine the Strength of a Potassium Cyanide Solution by a Decinormal Iodine Solution.—In titrating potassium cyanide with iodine, the reaction is:



$$253.70:65::0.012685:x. \quad x=0.00325.$$

Each cubic centimeter of a decinormal iodine solution corresponds to 0.00325 gram potassium cyanide. Use starch solution as an indicator. By multiplying the number of cubic centimeters of a decinormal iodine solution consumed before the blue color in the solution appears, by 0.00325, the weight of the potassium cyanide is found in the solution tested: from which the percentage strength can be calculated.

ATOMIC WEIGHTS.

ELEMENT.	SYMBOL.	ATOMIC WT. O = 16.
Aluminum.....	Al	27.1
Antimony.....	Sb	120.4
Arsenic.....	As	75.0
Barium.....	Ba	137.40
Bismuth.....	Bi	208.1
Boron.....	B	11.0
Bromine.....	Br	79.95
Cadmium.....	Cd	112.4
Carbon.....	C	12.0
Calcium.....	Ca	40.1
Chlorine.....	Cl	35.45
Chromium.....	Cr	52.1
Cobalt.....	Co	59.0
Copper.....	Cu	63.60
Fluorine.....	Fl	19.05
Gold.....	Au	197.2
Hydrogen.....	H	1.008
Iodine.....	I	126.85
Iron.....	Fe	55.9
Lead.....	Pb	206.92
Magnesium.....	Mg	24.3
Manganese.....	Mn	55.0
Mercury.....	Hg	200.0
Molybdenum.....	Mo	96.0
Nickel.....	Ni	58.70
Nitrogen.....	N	14.04
Oxygen.....	O	16.0
Phosphorus.....	P	31.0
Platinum.....	Pt	194.9
Potassium.....	K	39.11
Selenium.....	Se	79.2
Silicon.....	Si	28.4
Silver.....	Ag	107.92
Sodium.....	Na	23.05
Strontium.....	Sr	87.60
Sulphur.....	S	32.07
Tellurium.....	Te	127.7
Tin.....	Sn	119.0
Titanium.....	Ti	48.15
Zinc.....	Zn	65.4

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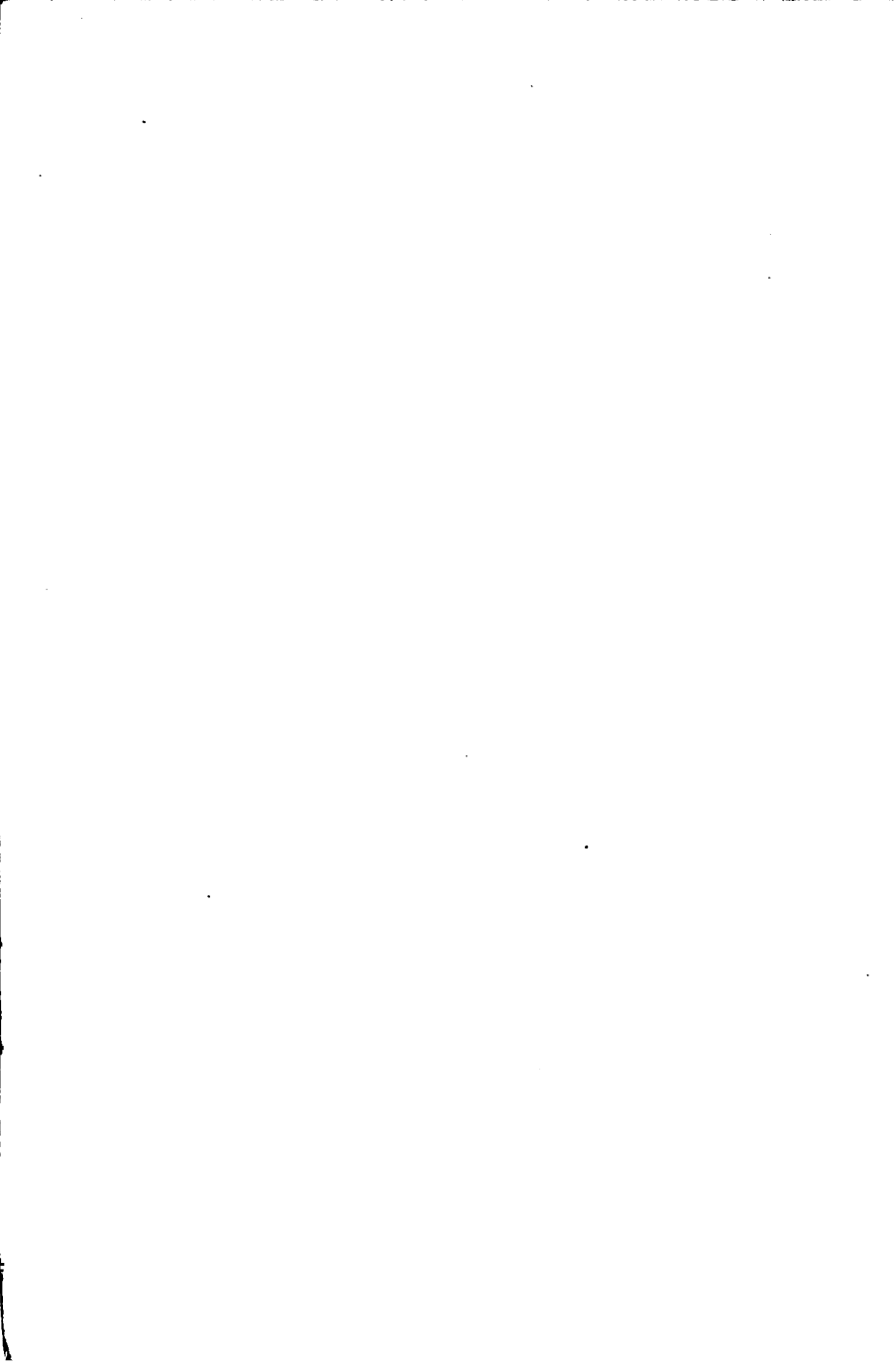
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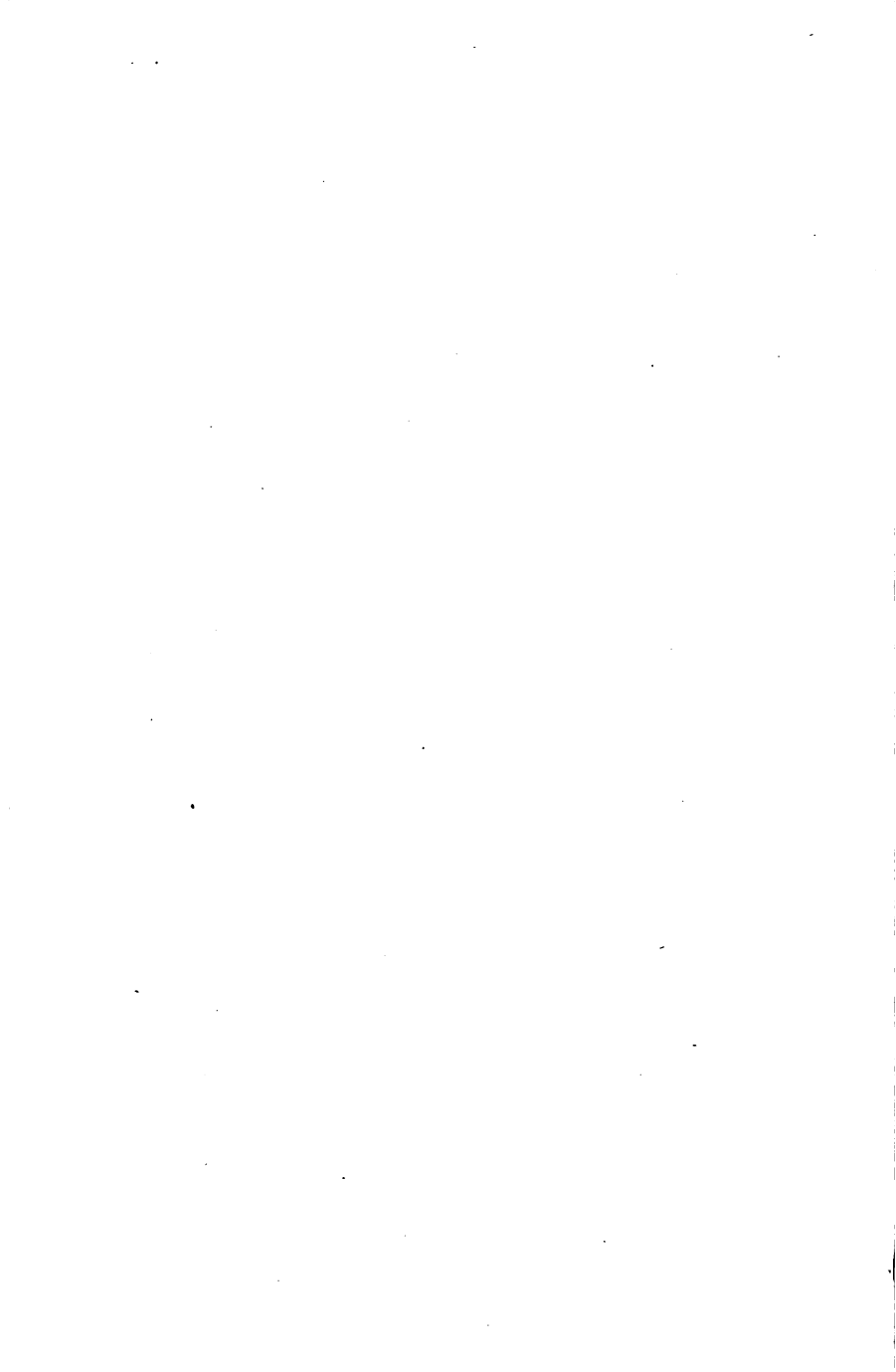
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